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(NASA-CR-150844) DEVELOPMENT OF LOW COST,
HIGH RELIABILITY SEALING TECHNIQUES FOR
HYBRID MICROCIRCUIT PACKAGES. PHASE 2,
SUPPLEMENT 1: MOISTURE PERMEATION OF
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DEVELOPMENT OF LOW COST, HIGH RELIABILITY SEALING TECHNIQUES FOR HYBRID MICROCIRCUIT PACKAGES

PHASE II - SUPPLEMENT NO. 1
MOISTURE PERMEATION OF
ADHESIVE-SEALED HYBRID MICROCIRCUIT PACKAGES

FINAL REPORT

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1.0 INTRODUCTION

1.1 STUDY BACKGROUND

As part of the continuing effort sponsored by NASA/MSFC to decrease costs and improve the reliability of hybrid microcircuits, Rockwell International Corporation was contracted to investigate epoxy adhesive sealing of packages to determine whether this approach is an acceptable alternate to metallurgical sealing. Adhesive sealing would provide many benefits over currently used metallurgical sealing. Adhesive sealing is a low temperature process (about 150°C), does not require the use of flux, eliminates the possibility of metal particle contamination from a weld sealing process, and permits easier delidding of packages for rework. In addition, adhesive sealing would result in considerable cost savings since it would allow the use of inexpensive, all-ceramic integral-lead packages instead of the currently used expensive gold-plated Kovar packages. Also, adhesive sealed packages are extensively used in commercial microelectronics and field data indicate that they have been performing reliably for years for those applications.

The initial study (Phase I) was started in June 1975 and addressed the following three tasks: (1) survey of representative hybrid manufacturers to assess the current use of adhesives for package sealing; (2) cost comparison of metallurgical versus adhesive sealing of packages; and (3) determination of the seal integrity of gold-plated Kovar butterfly-type packages sealed with selected adhesives after they had been subjected to selected MIL-STD-883A test environments. This study was documented in NASA Report CR-144339 issued in April 1976. Results showed that adhesive-sealed packages retained their seal integrity (as determined by the Seal Test specified in MIL-STD-883A, Method 1014.1, Test Conditions A₂ and C₁) after having been sequentially subjected to the following MIL-STD-883A test environments: Thermal Shock (15 cycles: 0°C, +100°C), Temperature Cycling (15 cycles: -65°C, +150°C), Mechanical Shock (five 1500g shock pulses in the Y₁ plane), and Constant Acceleration (10,000g's in the Y₁ plane). Gold-plated Kovar butterfly-type packages, 1.27 cm (1/2 inch) square, sealed with the epoxy film adhesives (Ablefilm 507, 529, and 550) and the paste adhesive (Epo-Tek H77) retained their seal

integrity after all tests, and similar 2.54 cm (1 inch) square packages retained their seal integrity after all tests except the 10,000 g's constant acceleration test. While these results were by no means considered to be sufficient to establish adhesives as suitable for sealing high reliability hybrid microcircuit packages, they were considered encouraging and indicative that adhesive package sealing warranted further evaluation.

As a result, a follow-on study (Phase II) was authorized in April 1976. This study consisted of three parts as follows:

- (1) Screening 10 selected adhesives on the basis of their ability to seal 2.54 cm (1 inch) square gold-plated Kovar packages that retain their seal integrity (as determined by the Seal Test specified in MIL-STD-883A, Method 1014.1, Test Conditions A₂ and C₁ and C₂) after individual exposures to the following four increasingly severe temperature-humidity environments:
 - (a) 50°C/60% RH for 10 days
 - (b) 60°C/98% RH for 10 days
 - (c) Moisture resistance test environment per Method 1004.1 of MIL-STD-883A
 - (d) 85°C/85% RH for 10 days
- (2) Screening the four best adhesives, as determined from the temperature-humidity screen, on the basis of their ability to seal 2.54 cm (1 inch) square gold-plated Kovar packages and 2.29 cm (0.9 inch) square ceramic packages that retain their seal integrity after both individual and sequential exposure to the following Class A test environments specified in Method 5004.2 of MIL-STD-883A:
 - (a) Thermal Shock: Method 1011.1, Test Condition C (15 cycles, -65°C to +150°C)
 - (b) Temperature Cycling: Method 1010.1, Test Condition C (15 cycles, -65°C to +150°C)
 - (c) Mechanical Shock: Method 2002.1, Test Condition B (5 shock pulses at 1500 g's in the Y₁ plane)
 - (d) Constant Acceleration: Method 2001.1, Test Condition A (5000 g's in the Y₁ plane)
 - (e) Temperature Aging corresponding to the temperature/time requirement associated with the burn-in test of Method 1015.1 (240 hours at 125°C)

- (3) Subjecting samples of the best adhesive/package combination, as identified by the MIL-STD-883A screen, to a 60°C/98% RH environment and continuously monitoring their moisture contents using Panametrics Aquamax-type moisture sensors to determine their susceptibility to moisture permeation.

This study is documented in Rockwell Report C76-597/201 issued in August 1977. Briefly, the results showed that while there are adhesives used in sealing packages that retain their seal integrity (i.e., pass the MIL-STD-883A Seal Test) after exposure to temperature-humidity environments including 10 days at 85°C/85% RH and all MIL-STD-883A, Class A test environments, the seals are not hermetic to moisture.

The investigation of the susceptibility of adhesive-sealed packages to moisture permeation, performed as the last task of this study, involved the testing of two ceramic packages sealed with Ablebond 789-1 and a seam-sealed gold-plated Kovar package serving as a control. These packages were subjected to the 60°C/98% RH environment for 15 days. The results obtained were as follows:

Seam-Sealed Gold-Plated Kovar Package. The moisture content remained constant throughout the 15-day exposure at a value that was less than 130 ppm_v and probably around 20 ppm_v indicating that this package had a good hermetic seal.

Ablebond 789-1 Adhesive-Sealed Ceramic Packages. The moisture content started out at the same value as that of the seam-sealed gold-plated Kovar package, increased very slowly to 1000 ppm_v after the first few days (2 days for one package and 3.75 days for the other), and then more rapidly and linearly for the remainder of the 15-day exposure. At the end of the 15-day exposure, one package contained approximately 14,600 ppm_v moisture and the other 19,000 ppm_v. This indicates that the moisture was diffusing into the packages rather than leaking into them and shows that adhesive-sealed ceramic packages are not hermetic.

1.2 SCOPE OF THE PRESENT STUDY

The investigation of the susceptibility of adhesive-sealed packages to moisture permeation performed during Phase II was limited to just two packages sealed with one type of adhesive. While the results obtained were considered to be significant, they also were considered to be preliminary and further investigation was recommended to substantiate them. As a result, the scope of work of the Phase II study was increased to include the determination of the moisture permeation of ceramic packages sealed with two different adhesives (Ablebond 789-1 and Epo-Tek H77) at the following four different temperature-humidity conditions: 25°C/98% RH; 50°C/60% RH; 60°C/98% RH; and 85°C/85% RH.

The present effort (Phase II, Supplement No. 1) consisted of testing a total of twenty packages — two ceramic packages sealed with each of the two different adhesives and one metallurgically sealed control package at each of the four different temperature-humidity environments.

2.0 TECHNICAL DISCUSSION

The work performed during this study is an extension of that performed as the last task of the Phase II Study discussed in Section 2.3 of the Phase II Final Report (Rockwell Report No. C76-597/201). The reader is referred to that report for descriptions of the equipment and procedures used.

The primary objective of this study was to determine the susceptibility of adhesive-sealed ceramic packages to moisture permeation during exposure to selected temperature-humidity environments by monitoring the outputs of solid-state moisture sensors sealed inside the packages. In addition to performing the effort required to accomplish this objective, other studies were performed to investigate the repeatability of the moisture sensor calibration curves and the effect of exposure to pre-sealing bake-out conditions and curing time-temperature schedules on the moisture sensor calibration curves.

2.1 GENERAL DISCUSSION

Two adhesives, Ablebond 789-1 and Epo-Tek H77, were evaluated as package sealants at temperature-humidity conditions of 25°C/98% RH, 50°C/60% RH, 60°C/98% RH, and 85°C/85% RH using Panametrics Mini-Mod-A moisture sensors. Five packages were tested at each of the four temperature-humidity conditions — two ceramic packages sealed with each of the two adhesives being evaluated, and one seam-sealed gold-plated Kovar package to provide a control.

2.1.1 The Panametrics Mini-Mod-A Moisture Sensor

At the recommendation of Panametrics, the Mini-Mod-A was used as the moisture sensor rather than the Aquamax-type previously used. A photograph of this sensor is shown in Figure 1. The dimensions of the sensor chips are 0.152 cm (60 mils) square and the active areas are 0.0087 cm^2 (1.35 mil^2). The construction of this sensor is apparently the same as that of the Aquamax (reference 1), but some proprietary processing changes must have been made because the vendor claims this sensor has greater stability than the Aquamax. According to the vendor's literature, the Mini-Mod-A may be baked at 150°C in vacuum as

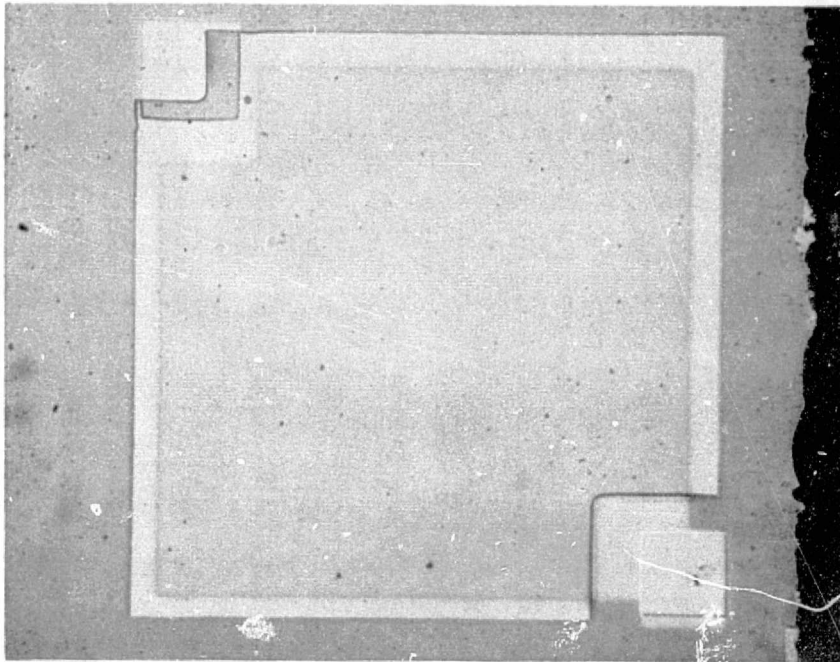
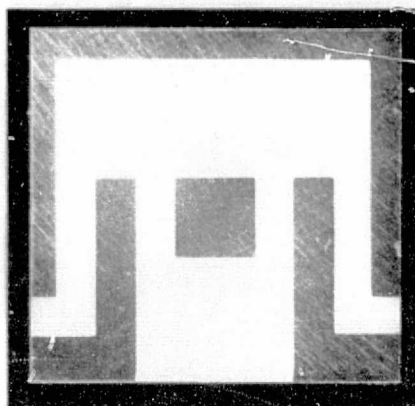


Figure 1. Photograph of a Panametrics
Mini-Mod-A Moisture Sensor
(~70X)

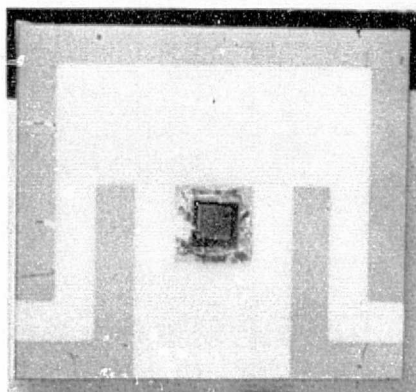
long as 30 hours, and may be mounted with epoxy adhesives that can be cured at 150°C in two hours or less. It is reported that the sensors can be exposed to these conditions without changes in the calibration curves as supplied with the sensors. An accuracy of $\pm 25 \text{ ppm}_v$ or $\pm 25\%$ in ppm_v (whichever is greater) in the range of 100 ppm_v to $10,000 \text{ ppm}_v$ is quoted. The recommended measurement temperature is $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$, as for the Aquamax.

2.1.2 Sensor Mounting

The thin film gold metallized ceramic carrier substrates for the moisture sensors were redesigned to accommodate the Mini-Mod-A. Photographs of the carrier substrate and a substrate with an attached Mini-Mod-A moisture sensor are shown in Figure 2. Use of these carrier substrates was necessary to permit multiple mounting and removal of the moisture sensors without damaging them. The substrates with the moisture sensors attached, rather than the moisture sensors themselves, were the units that were mounted and removed as required during the course of the investigation. The sensors were attached to the carrier substrates with pure indium solder (melting temperature 200°C).



(a) Carrier Substrate
(~4X)



(b) Substrate With Mini-Mod-A
Moisture Sensor Attached

Figure 2. Thin Film Gold Metallized
Ceramic Carrier Substrate

2.1.3 Ceramic Package Bases

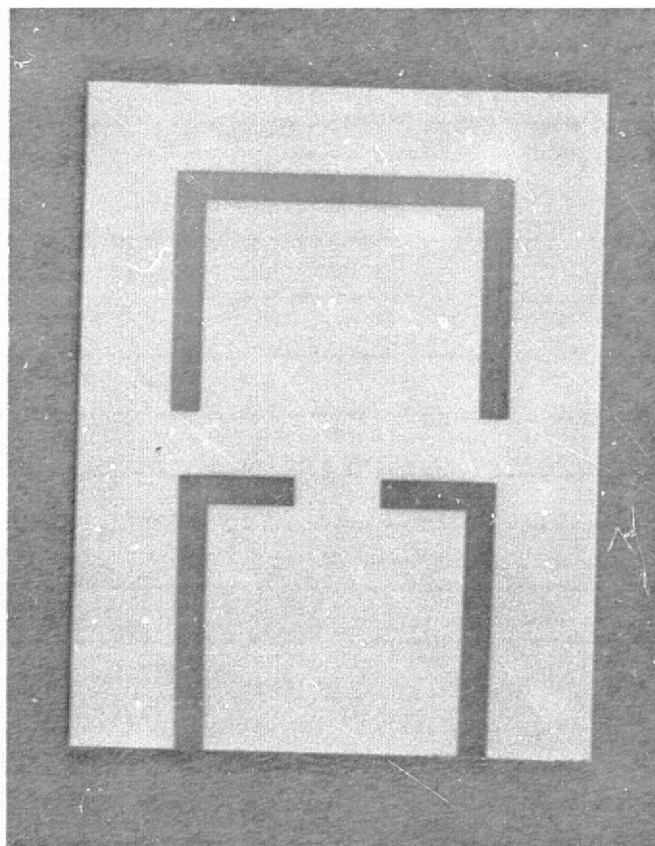
The thin film gold metallized ceramic package base was redesigned with increased spacing between the output lines to decrease the possibility of interline leakage current that might occur when the packages are exposed to the high relative humidities required in this study. Photographs of a ceramic package base and one with a carrier substrate (and moisture sensor) attached are shown in Figure 3.

2.1.4 Multiplexer for Measurement of Multiple Sensors

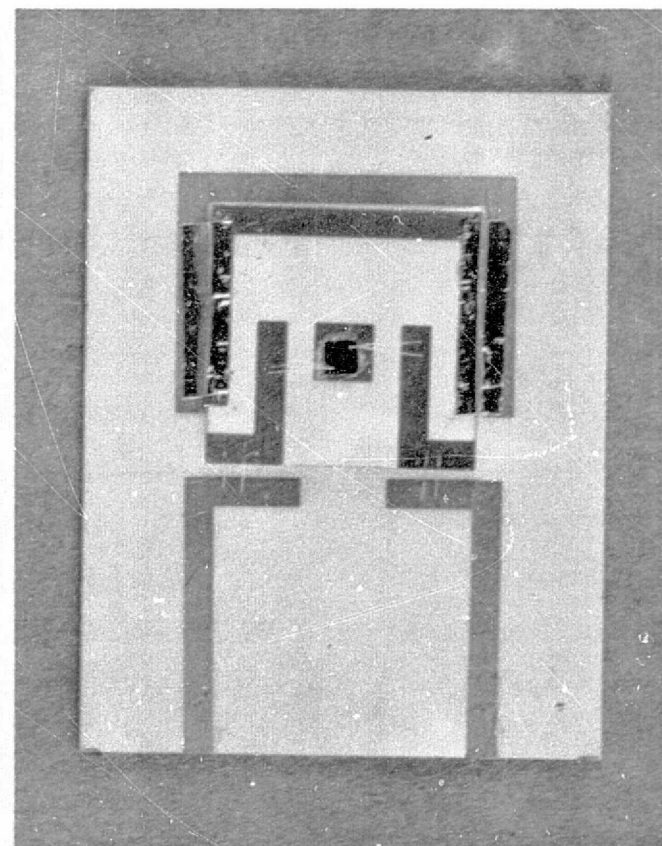
A multiplexer capable of sequentially monitoring the outputs of the moisture sensors in all of the packages being tested at the same time was designed and fabricated. This multiplexer accommodated six inputs, sequencing through them once an hour (i.e.; each sensor was sampled once each hour for a period of 10 minutes). This permitted continuous unattended monitoring of the outputs of the moisture sensors in the five packages being tested as well as the Control Unit zero output. External and internal views of the multiplexer are given in Figures 4 and 5, respectively.

2.2 REPEATABILITY OF MOISTURE SENSOR CALIBRATION CURVES

To determine the repeatability of the moisture sensor calibration curves, the same five moisture sensors were calibrated on three consecutive days. Prior to each calibration, the chamber (with sensors) was purged overnight with dry nitrogen. The calibration runs were made at 50°C at increasing moisture levels of 200, 1000, 2600, 6000, and 10,000 ppm_v (dew points of -36, -20, -10, 0, and +7°C, respectively). Results are graphed in Figures 6 through 10. Repeatability was reasonably good but not excellent. For example, for approximate moisture values of 250, 1000, 5000 and 9000 ppm_v, the values obtained from the worst case curves (Sensor No. 7) vary from 210 to 270, 800 to 1125, 4600 to 5250, and 8500 to 9500 ppm_v. This indicates variations of ± 12.5 , ± 16.9 , ± 6.6 , and $\pm 5.6\%$ which are well within the $\pm 25\%$ accuracy specified by the manufacturer.



(a) Base for Ceramic Package



(b) Ceramic Package Base With Carrier Substrate
(and Moisture Sensor) Attached

Figure 3. Thin Film Gold Metallized Ceramic Package Base

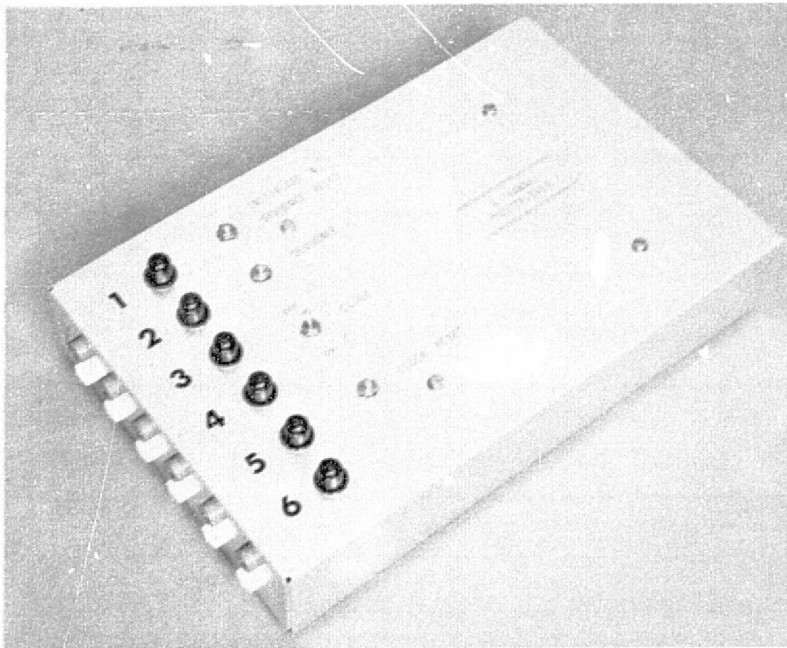


Figure 4. External View of Six-Channel Multiplexer

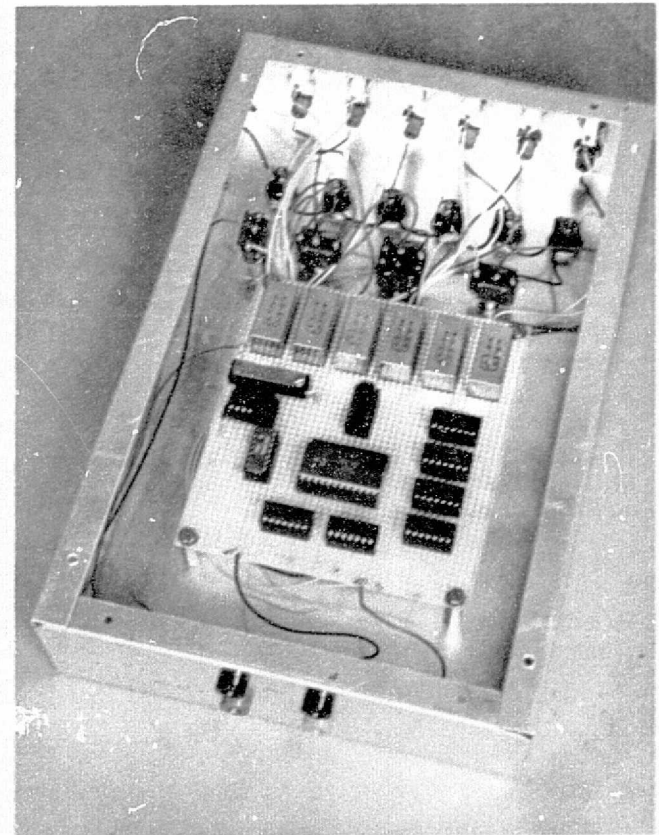


Figure 5. Internal View of Six-Channel Multiplexer

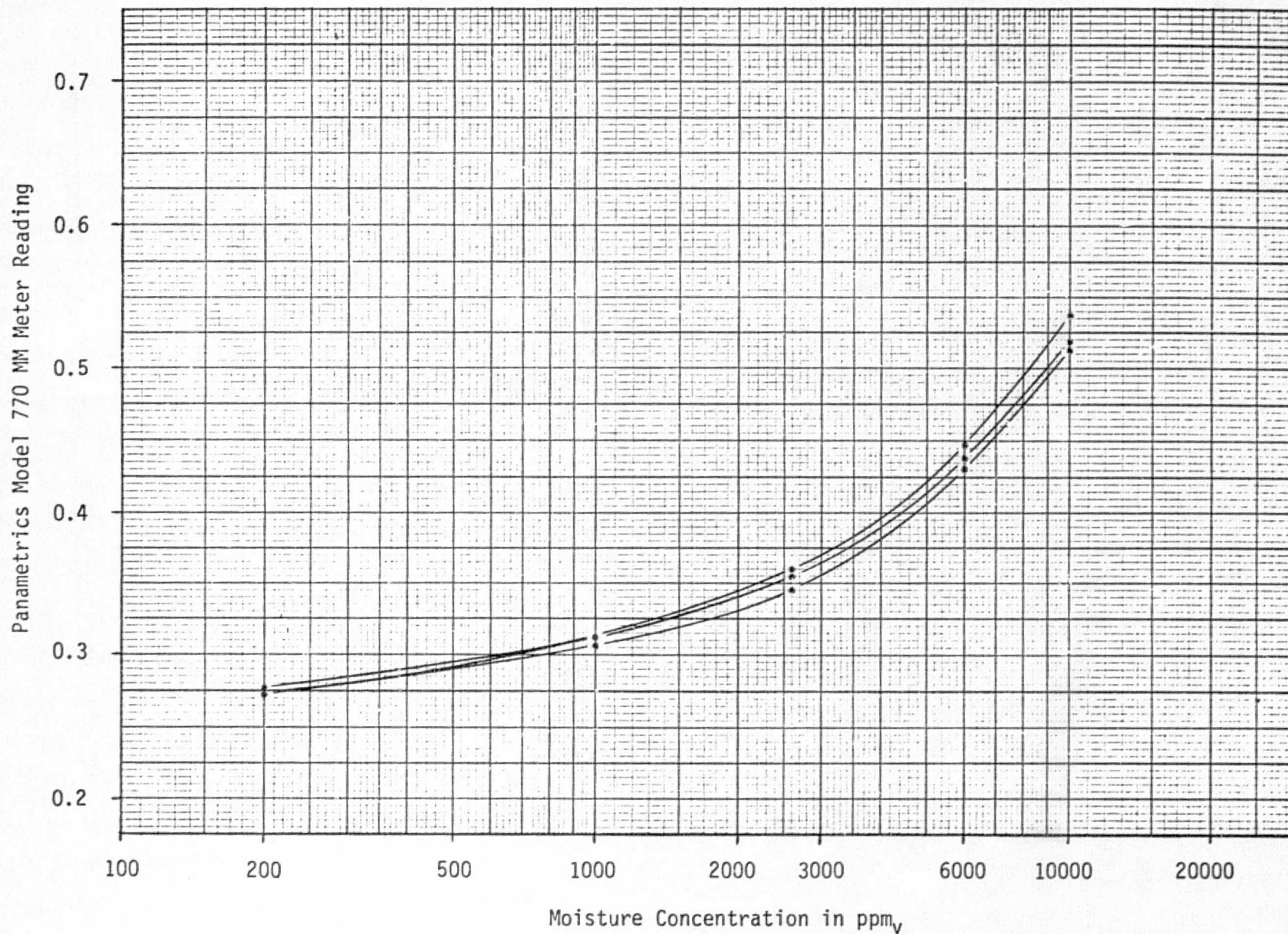


Figure 6. Calibration Curves for Mini-Mod-A Moisture Sensor No. 6 at 50°C

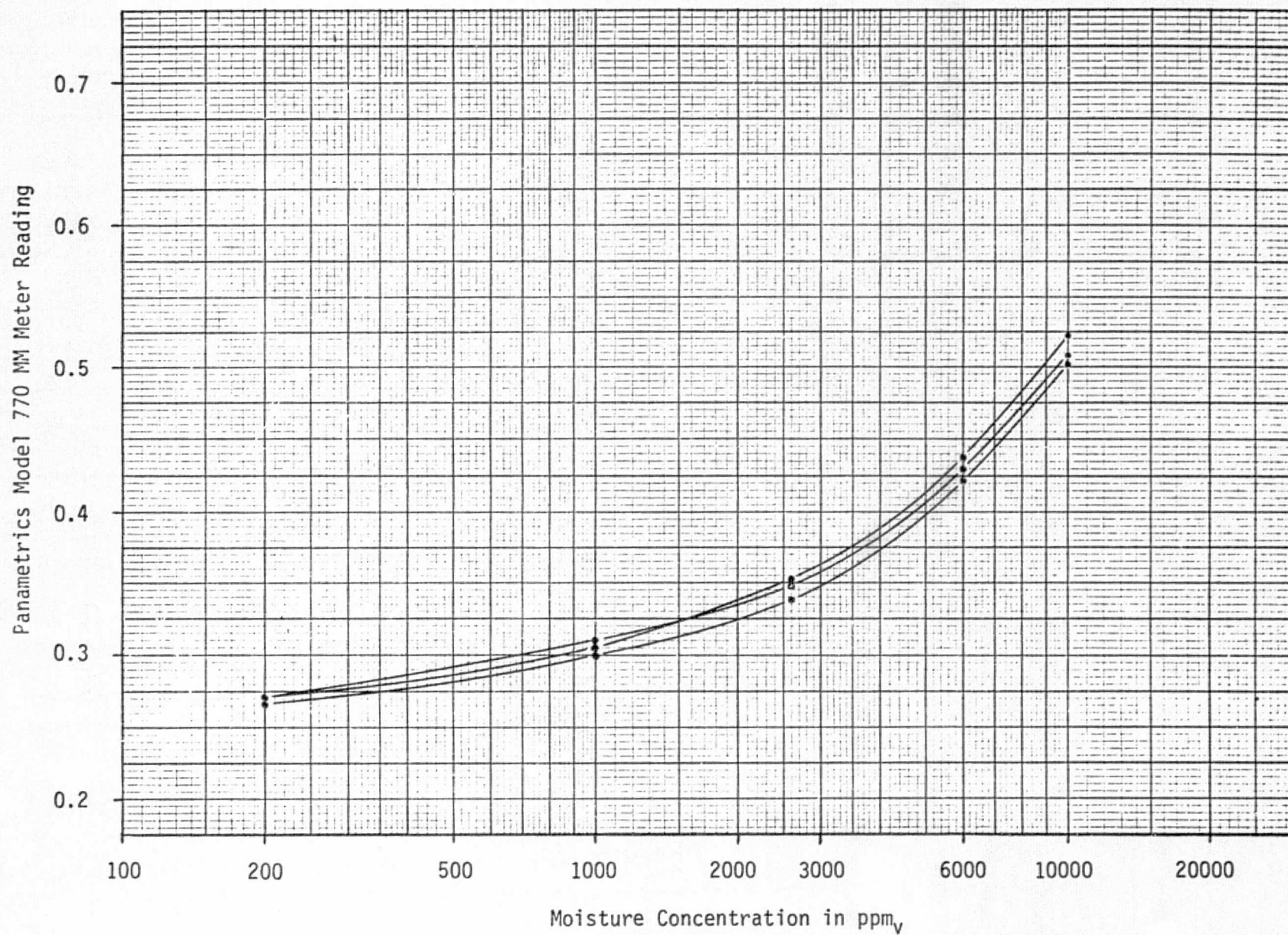


Figure 7. Calibration Curves for Mini-Mod-A Moisture Sensor No. 7 at 50°C

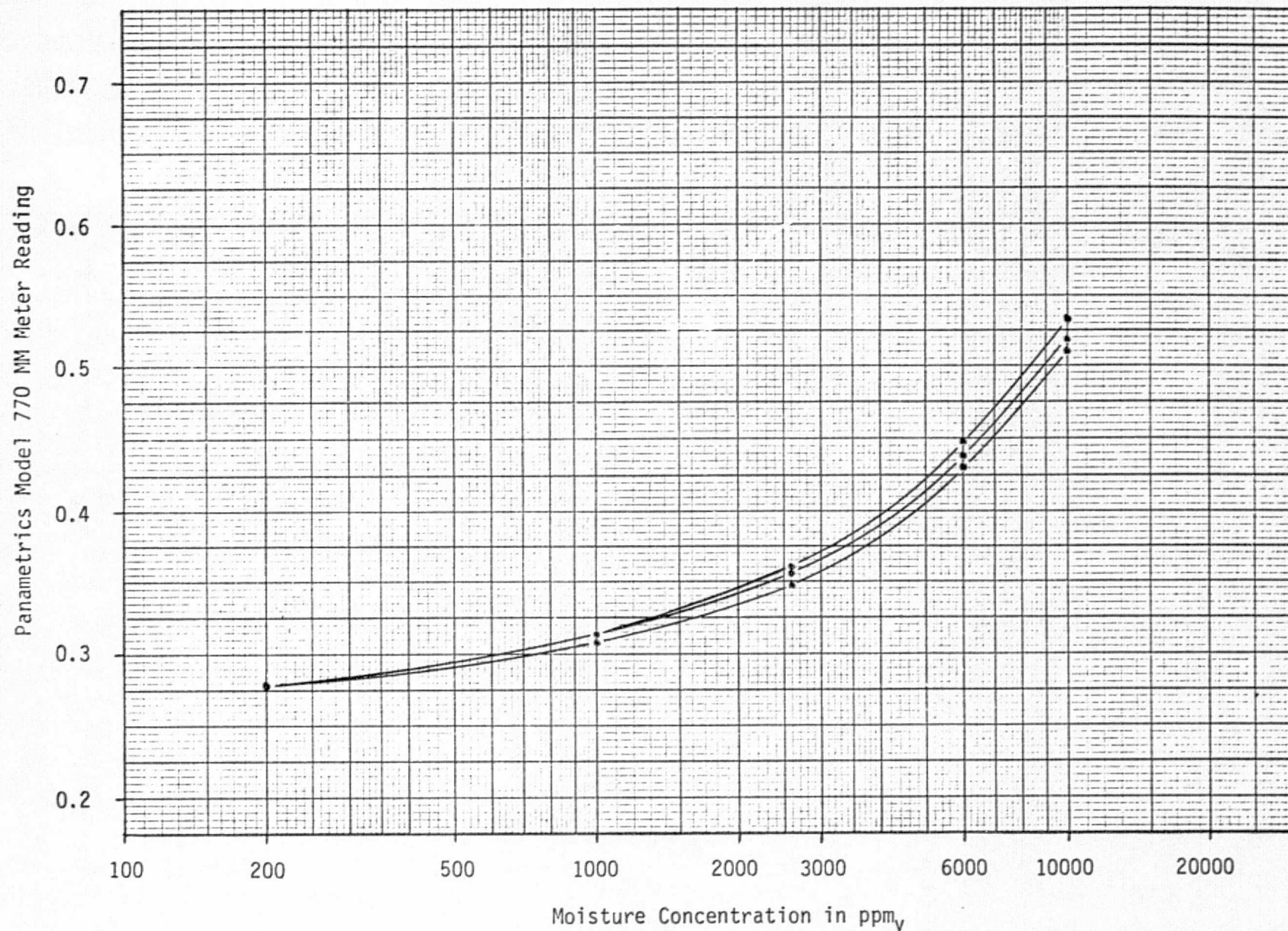


Figure 8. Calibration Curves for Mini-Mod-A Moisture Sensor No. 8 at 50°C

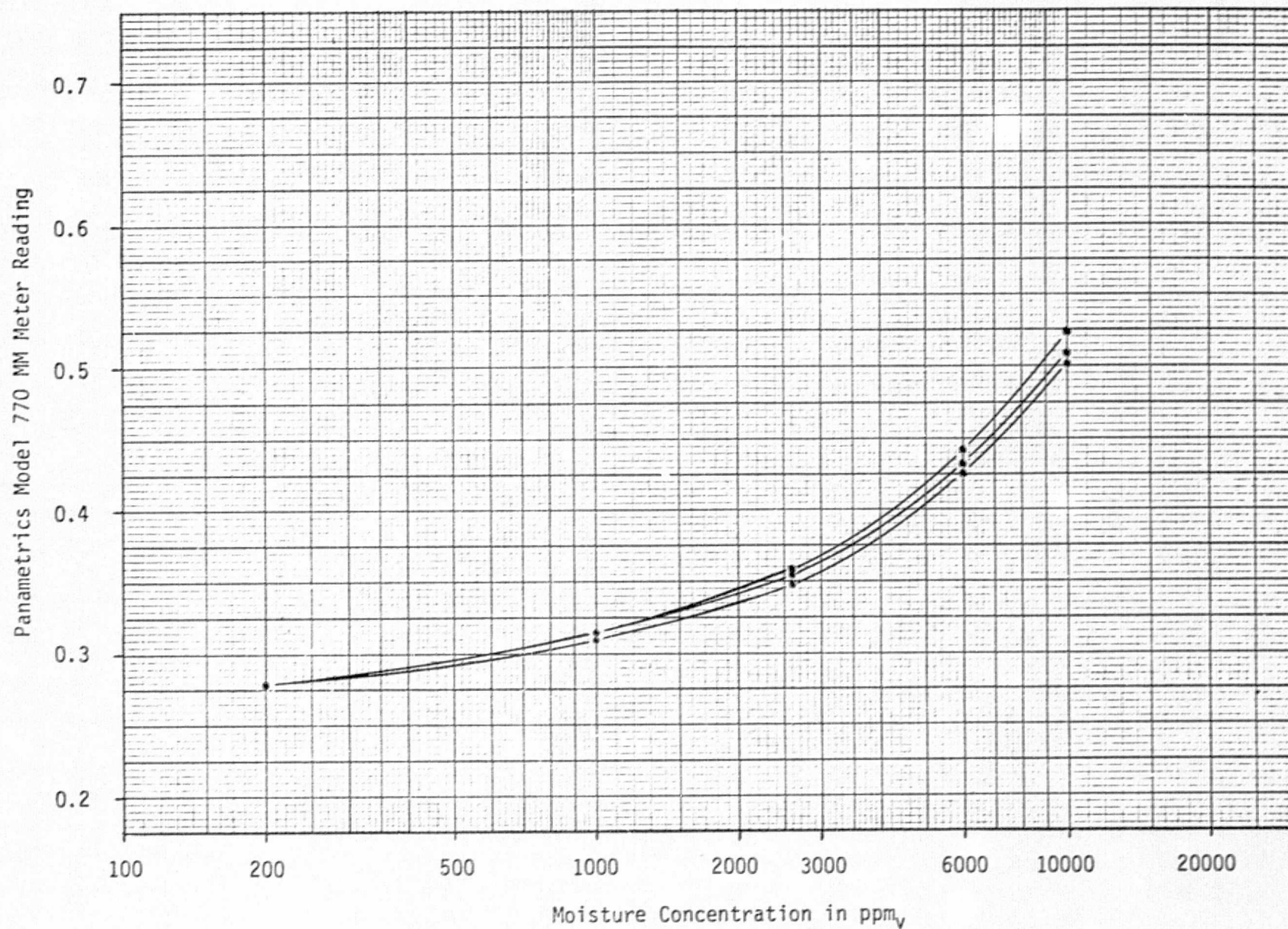


Figure 9. Calibration Curves for Mini-Mod-A Moisture Sensor No. 9 at 50°C

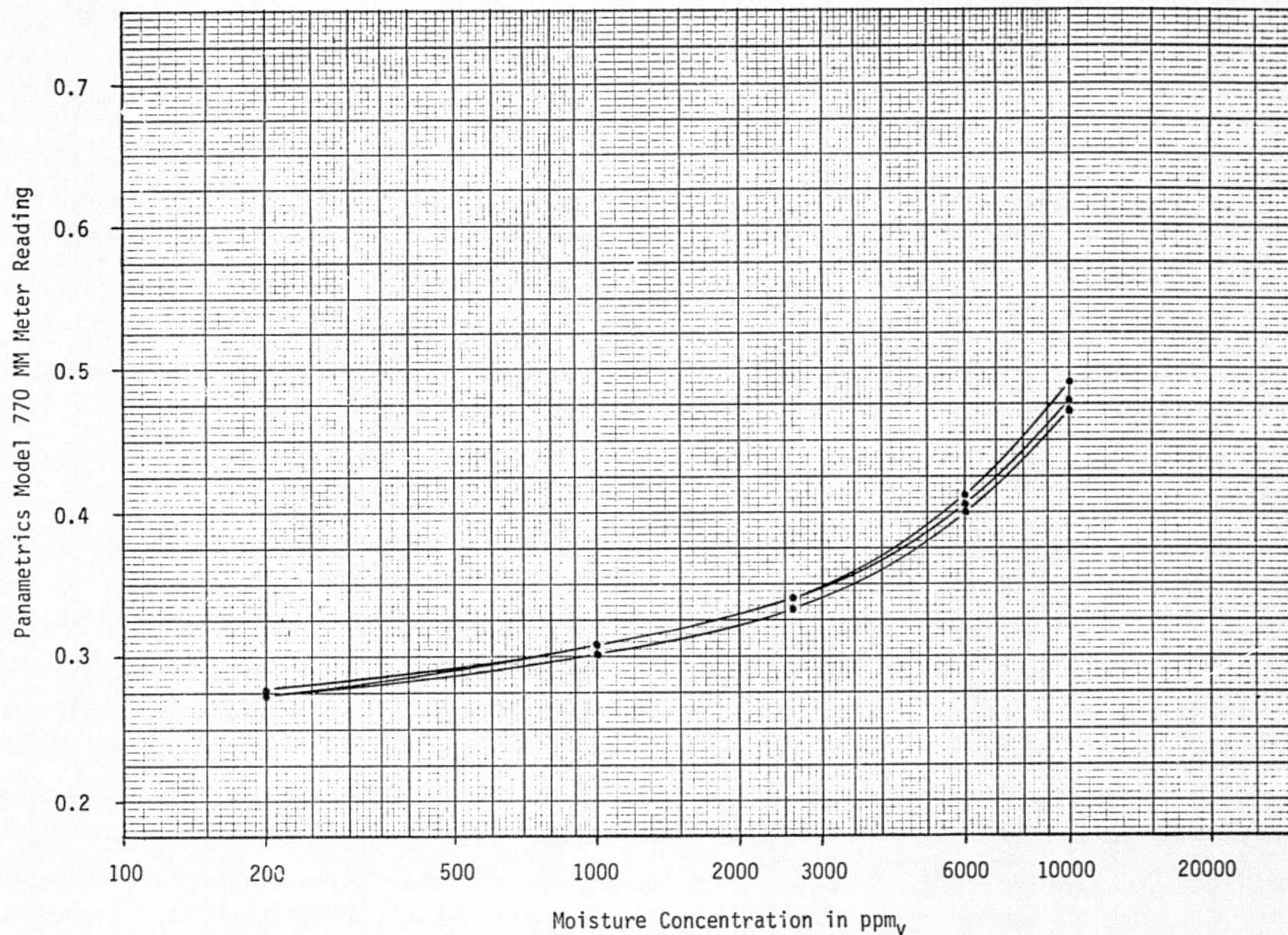


Figure 10. Calibration Curves for Mini-Mod-A Moisture Sensor No. 10 at 50°C

2.3 TESTING PROCEDURE AND RESULTS OBTAINED AT DIFFERENT TEMPERATURE-HUMIDITY EXPOSURES

2.3.1 Package Preparation and Testing

Two ceramic packages sealed with each of the two adhesives and one seam-sealed gold-plated Kovar package (control) were prepared for exposure to each of the four temperature-humidity environments. The packages were assembled and sealed as discussed in Section 2.3.8.1 of the Phase II Final Report. Essential points that should be emphasized here are the following:

- (1) The gold-plated Kovar packages were vacuum baked for four hours at 135°C immediately prior to sealing and were seam-sealed in an attached dry nitrogen glove box without exposure to any other ambient. These packages were brazed rather than welded; i.e., gold-tin preforms were used.
- (2) The adhesive-sealed ceramic packages were assembled in room ambient and cured in a dry nitrogen environment with a clamping pressure of approximately $7.0 \times 10^4 \text{ N/m}^2$ (10 psi). The packages sealed with Able-bond 789-1 were cured for 2 hours at 170°C and those sealed with Epo-Tek H77 were cured for 30 minutes at 150°C. After curing, the packages were removed from the curing oven and transferred directly to the attached dry nitrogen glove box, the vent holes were sealed with Epo-Tek H77, and the packages replaced in the dry nitrogen oven and cured for 30 minutes at 150°C. To ensure that the vent holes were sealed, this process was repeated.
- (3) Moisture sensor readings were then taken and the packages were fine and gross leak tested per MIL-STD-883A, Method 1014.1, Test Conditions A₂ and C₁. Results of the leak tests were recorded and moisture sensor readings were again taken to establish the initial integrity of the packages.

2.3.2 Test Procedure

For each temperature-humidity exposure the procedure followed was:

- (1) The sensors were calibrated at exposure temperatures of 25, 50, 60, or 85°C over a moisture range of 200 to 10,000 ppm_v and then sealed in the packages as described above.

- (2) The packages were exposed to temperature-humidity environments of 25°C/98% RH, 50°C/60% RH, 60°C/98% RH, or 85°C/85% RH and the sensor outputs monitored and recorded.
- (3) The packages were leak tested per MIL-STD-883A, Method 1014.1, Test Conditions A₂ and C₁ to verify retention of their seal integrity.
- (4) The sensors were removed from the packages and recalibrated at the exposure temperature.
- (5) The sensor output readings taken during the exposure were interpreted in ppm_v using the post-exposure calibration curves and plotted versus exposure time.

Detailed presentation of the data and discussion of the results follows.

2.3.3 25°C/98% RH Exposure Results

Moisture sensor readings taken during this exposure are given in Table 1. Due to the low rate of moisture permeation for most of the packages at this condition, exposure was continued for 40 days (approximately 1000 hours). Post-exposure leak rates of the packages are given in Table 2 along with those measured prior to exposure. Comparison of these leak rates shows that the integrity of the package seals were not affected by the 40-day 25°C/98% RH exposure. The post-exposure calibration curves for the moisture sensors are given in Figures 11 through 15. The initial calibration curves are also included for comparison. It should be noted that the shifts in the calibration curves are not uniform even for those sensors exposed to the same curing time-temperature schedules (Table 3).

Conversion of the sensor readings of Table 1 to moisture content in ppm_v based on the post-exposure calibration curves is given in Table 4. These results are graphed in Figure 16. Moisture permeation into the seam-sealed gold-plated Kovar package and into the two ceramic packages sealed with Ablebond 789-1 was very slow. The final moisture content of the seam-sealed package after 40 days exposure was 360 ppm_v and those of the two Ablebond 789-1 sealed packages were 330 and 650 ppm_v, respectively. Moisture permeated much more rapidly into the two ceramic packages sealed with Epo-Tek H77. For one of these packages, the moisture content reached 12,200 ppm_v by the end of the

TABLE 1. MOISTURE SENSOR READINGS DURING 25°C/98% RH EXPOSURE

Exposure (Days)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Gold-Plated Kovar Package Seam-Sealed
	Sensor No. 1	Sensor No. 2	Sensor No. 3	Sensor No. 4	Sensor No. 5
0	0.222	0.220	0.341	0.300	0.250
1	0.223	0.221	0.382	0.301	0.251
2	0.223	0.221	0.420	0.302	0.252
4	0.225	0.223	0.482	0.310	0.255
5	0.227	0.224	0.511	0.311	0.257
6	0.228	0.225	0.541	0.313	0.258
7	0.229	0.227	0.572	0.318	0.258
8	0.229	0.227	--	0.319	0.254
9	0.229	0.228	--	0.321	0.255
12	0.230	0.229	--	0.332	0.257
13	0.230	0.229	--	0.338	0.258
14	0.230	0.230	--	0.341	0.257
15	0.231	0.230	--	0.348	0.259
16	0.231	0.231	--	0.350	0.258
18	0.232	0.232	--	0.360	0.259
20	0.232	0.233	--	0.368	0.259
21	0.232	0.237	--	0.371	0.259
22	0.235	0.238	--	0.375	0.259
23	0.237	0.239	--	0.380	0.260
26	0.239	0.240	--	0.390	0.261
27	0.239	0.241	--	0.392	0.263
28	0.239	0.241	--	0.397	0.271
29	0.240	0.242	--	0.400	0.272
30	0.240	0.243	--	0.403	0.269
33	0.241	0.249	--	0.412	0.272
34	0.242	0.249	--	0.418	0.273
35	0.242	0.250	--	0.420	0.275
36	0.243	0.251	--	0.423	0.278
37	0.245	0.251	--	0.428	0.279
40	0.249	0.258	--	0.438	0.281

TABLE 2. LEAK RATES OF PACKAGES EXPOSED TO 25°C/98% RH

Package Number	Description	Air Equivalent (atm cc/sec)	
		Prior to Exposure	After Exposure
1	Ceramic Packages Sealed With Ablebond 789-1	1.5×10^{-7}	3.3×10^{-7}
2		2.8×10^{-7}	3.0×10^{-7}
3	Ceramic Packages Sealed With Epo-Tek H77	3.0×10^{-7}	3.4×10^{-7}
4		3.0×10^{-7}	2.6×10^{-7}
5	Seam-Sealed Gold-Plated Kovar Package	2.4×10^{-9}	3.4×10^{-9}

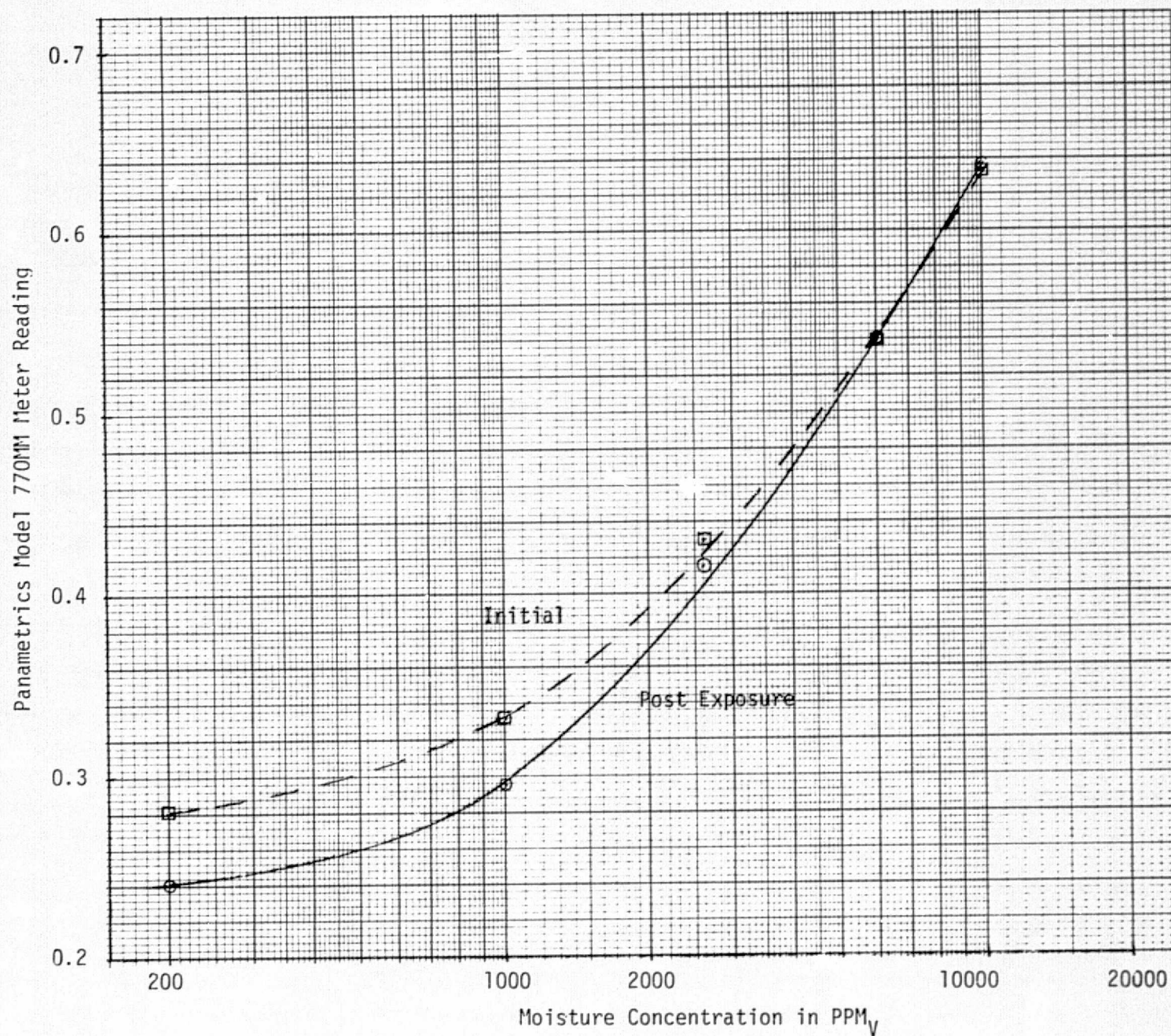


Figure 11. Initial and Post Exposure (25°C/98% RH) Calibration Curves for Moisture Sensor No. 1 at 25°C

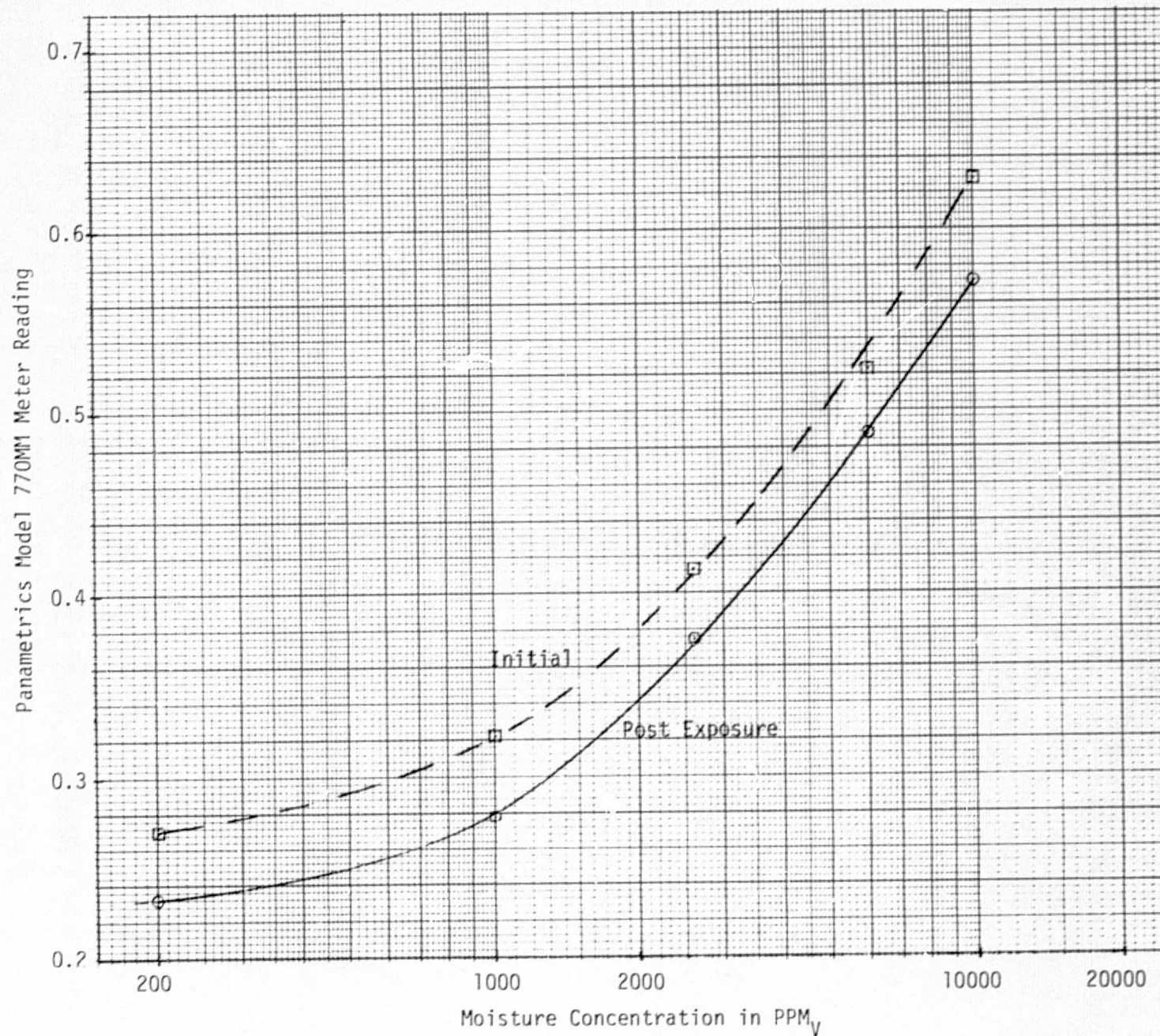


Figure 12. Initial and Post Exposure (25°C/98% RH) Calibration Curves for Moisture Sensor No. 2 at 25°C

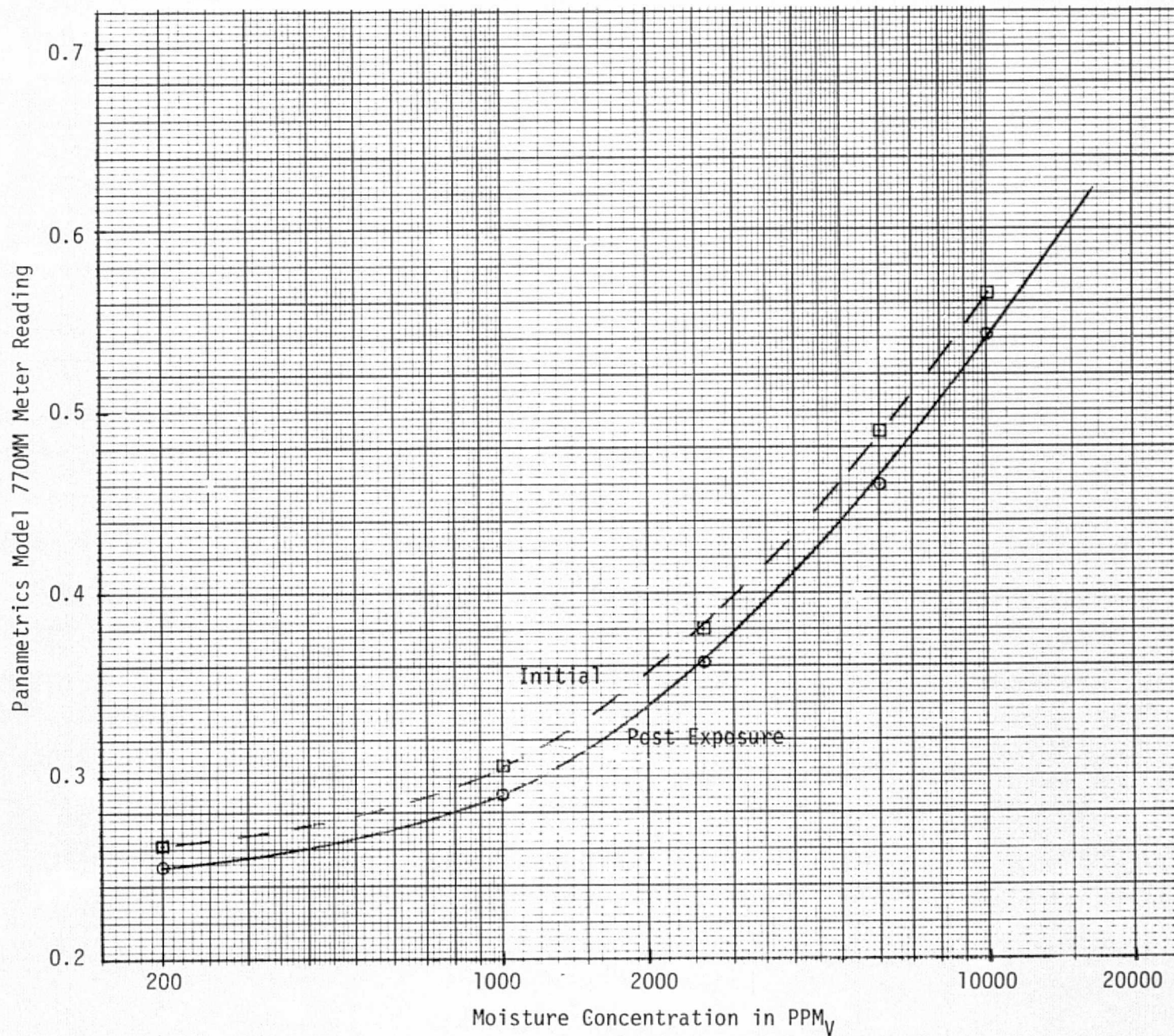


Figure 13. Initial and Post Exposure (25°C/98% RH) Calibration Curves for Moisture Sensor No. 3 at 25°C

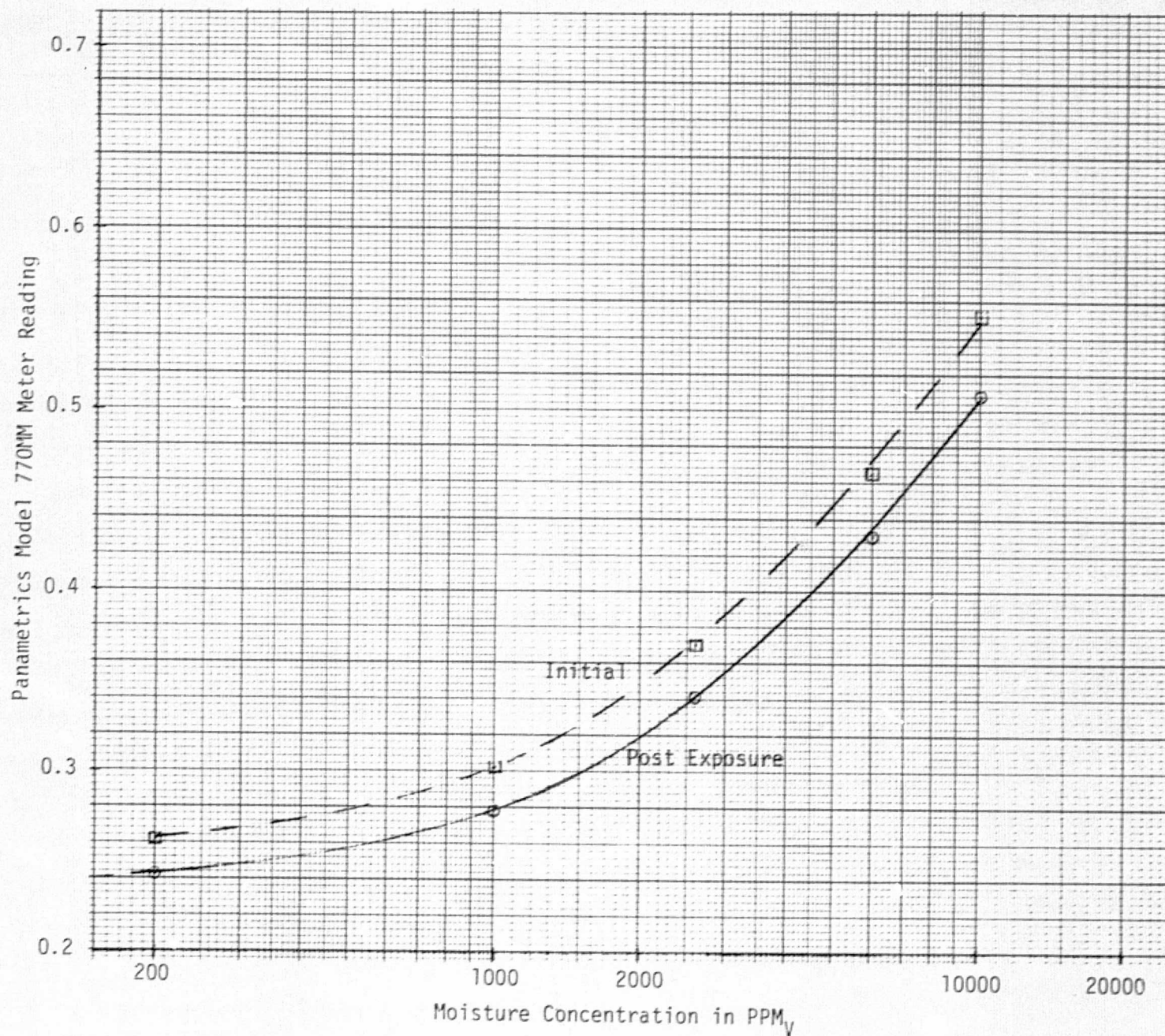


Figure 14. Initial and Post Exposure (25°C/98% RH) Calibration Curves for Moisture Sensor No. 4 at 25°C

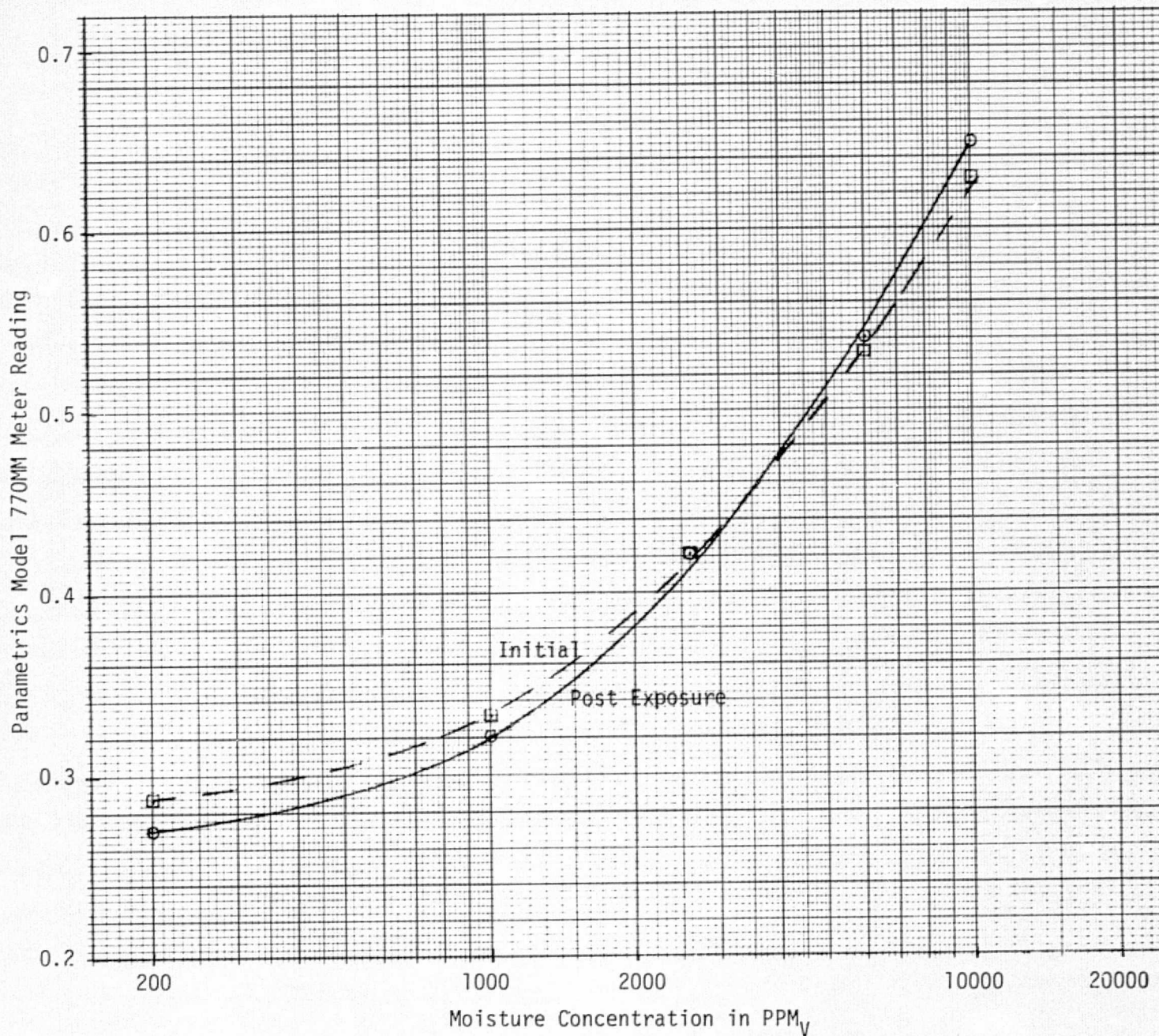


Figure 15. Initial and Post Exposure (25°C/98% RH) Calibration Curves for Moisture Sensor No. 5 at 25°C

TABLE 3. TEMPERATURE EXPOSURES EXPERIENCED BY SENSORS

Sensor No.	Sealing Method	Temperature Exposure
1 & 2	Sealed With Ablebond 789-1 Epoxy	2 Hours at 170°C plus 1 Hour at 150°C
3 & 4	Sealed With Epo-Tek H77 Epoxy	1-1/2 Hours at 150°C
5	Seam-sealed	Vacuum Baked for 4 Hours at 135°C

TABLE 4. MOISTURE CONTENT OF PACKAGES IN PPM_V
DURING 25°C/98% RH EXPOSURE

Exposure (Days)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Gold-Plated Kovar Package Seam-Sealed
	Package No. 1 Sensor No. 1	Package No. 2 Sensor No. 2	Package No. 3 Sensor No. 3	Package No. 4 Sensor No. 4	Package No. 5 Sensor No. 5
0	<100	<100	2050	1530	<100
1	<100	<100	3100	1560	<100
2	<100	<100	4250	1590	<100
4	<100	<100	6800	1780	<100
5	<100	<100	8200	1800	<100
6	<100	<100	9900	1860	<100
7	<100	<100	12,200	1980	<100
8	<100	<100		2020	<100
9	<100	100		2050	<100
12	<100	130		2350	<100
13	<100	130		2500	<100
14	<100	160		2570	<100
15	<100	160		2800	<100
16	<100	180		2850	<100
18	<100	200		3150	<100
20	<100	220		3400	<100
21	<100	290		3500	<100
22	100	300		3600	<100
23	140	320		3800	<100
26	175	340		4150	<100
27	175	360		4200	<100
28	175	360		4400	210
29	200	375		4500	220
30	200	390		4600	180
33	210	500		5000	220
34	220	500		5200	230
35	220	520		5300	270
36	230	530		5400	310
37	260	530		5700	330
40	330	650		6100	360

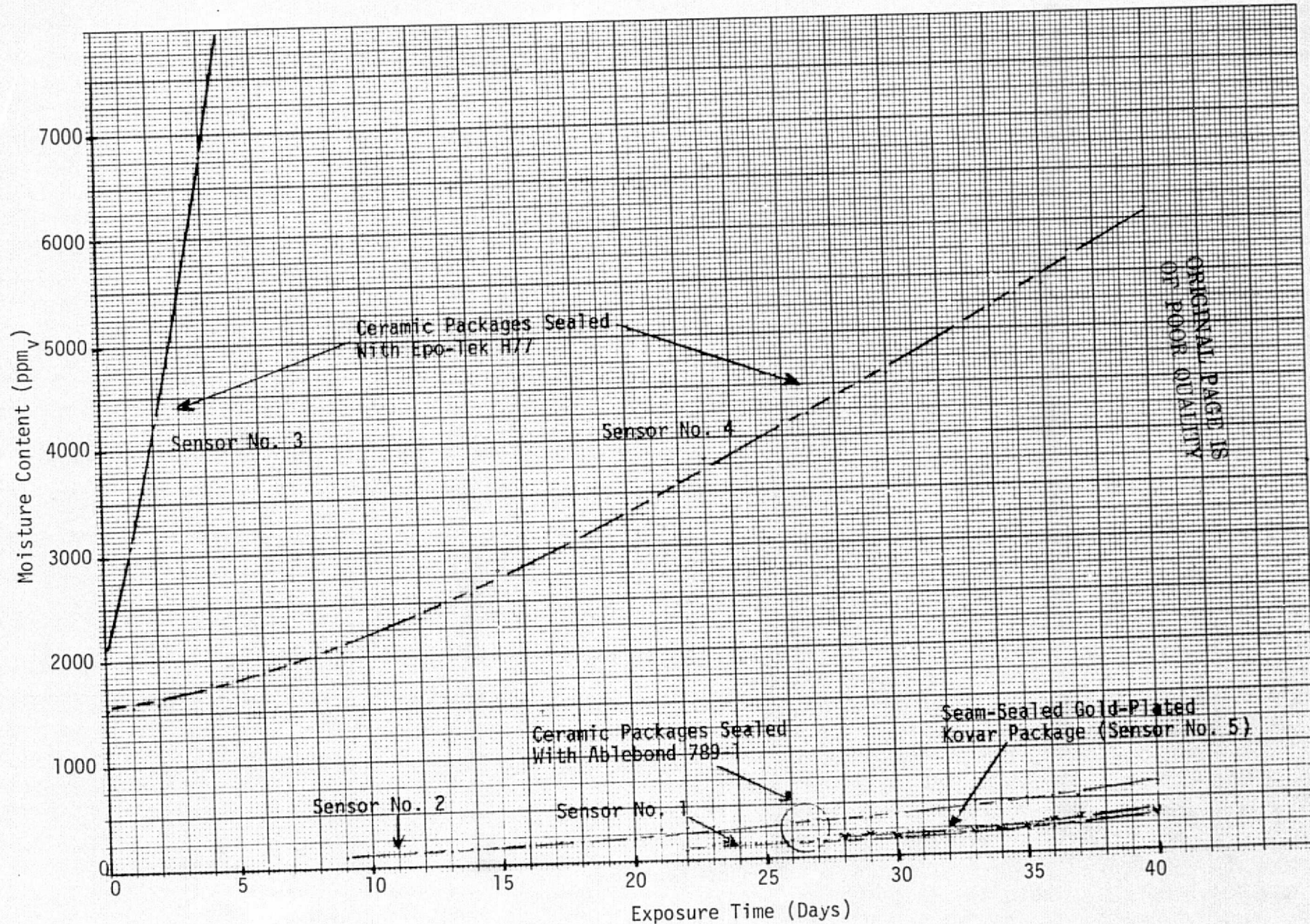


Figure 16. Moisture Permeation Into Packages Exposed to 25°C/98% RH

seventh day and testing was terminated. For the other, it reached 6100 ppm_v by the end of the 40-day exposure. Although the initial (pre-exposure) measured leak rates for these two packages were identical (3.0×10^{-7} atm cc/sec air equivalent) and the post-exposure leak rates were essentially the same (3.4 and 2.6×10^{-7} atm cc/sec air equivalent), Package No. 3 apparently was a leaker.

The final permeation rates as determined by direct scaling from the graphs were 21 ppm_v/day for the seam-sealed gold-plated Kovar package, 19 to 26 ppm_v/day for the Ablebond 789-1 adhesive-sealed ceramic packages, and 146 ppm_v/day for the good Epo-Tek H77 adhesive-sealed ceramic package.

2.3.4 50°C/60% RH Exposure Results

The moisture sensor readings for the 50°C/60% RH exposure are given in Table 5. The two ceramic packages sealed with Ablebond 789-1 (containing Sensors Nos. 6 and 7) and the seam-sealed gold-plated Kovar package (containing Sensor No. 10) were exposed for 21 days. However, exposure of the two ceramic packages sealed with Epo-Tek H77 (containing Sensors Nos. 8 and 9) was terminated after 11 days because the sensor readings indicated that the moisture contents of these packages were approaching 15,000 ppm_v, the upper limit that Panametrics recommends for these sensors. According to Panametrics, prolonged exposure to moisture concentrations exceeding this level can damage the sensors. Since in the present case the sensors must be recalibrated to interpret the data, this could not be risked.

The leak test results after exposure are given in Table 6 along with those obtained prior to exposure. As can be seen, the leak rates of the packages were not significantly changed as a result of the extended exposure to the 50°C/60% RH environment. Difficulty was experienced in opening the ceramic packages to remove the sensors. The method finally used consisted of carefully cracking the package lids. However, prior to this, two of the packages (those containing Sensors Nos. 6 and 8) were heated on a hot-plate to approximately 180°C while attempts were made to pry the lids off. When these two sensors were tested prior to starting calibration, it was found that they were electrically open. Subsequent visual inspection and electrical tests were unsuccessful in isolating their failure. However, unquestionably, the sensors failed due to excessive exposure to the 180°C temperature since the other three sensors (Nos. 7, 9 and 10) that had not been so exposed were functional.

TABLE 5. MOISTURE SENSOR READINGS DURING 50°C/60% RH EXPOSURE

Exposure (Days)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Gold-Plated Kovar Package Seam-Sealed
	Sensor No. 6	Sensor No. 7	Sensor No. 8	Sensor No. 9	Sensor No. 10
0	0.228	0.223	0.248	0.242	0.262
1	0.228	0.226	0.250	0.247	0.268
2	0.230	0.230	0.260	0.253	0.275
3	0.235	0.235	0.291	0.279	0.283
4	0.238	0.240	0.327	0.309	0.292
7	0.248	0.255	0.449	0.407	0.318
8	0.252	0.261	0.491	0.443	0.322
9	0.258	0.268	0.525	0.478	0.330
10	0.260	0.272	0.551	0.505	0.339
11	0.262	0.279	0.574	0.529	0.348
14	0.273	0.297			0.370
15	0.278	0.302			0.378
16	0.281	0.310			0.388
17	0.285	0.317			0.392
18	0.290	0.322			0.398
21	0.302	0.342			0.418

TABLE 6. LEAK RATES OF PACKAGES EXPOSED TO 50°C/60% RH

Package Number	Description	Air Equivalent (atm cc/sec)	
		Prior to Exposure	After Exposure
6	Ceramic Packages Sealed With Ablebond 789-1	1.9×10^{-7}	1.4×10^{-7}
7		2.1×10^{-7}	3.6×10^{-7}
8	Ceramic Packages Sealed With Epo-Tek H77	2.5×10^{-7}	3.0×10^{-7}
9		2.1×10^{-7}	1.5×10^{-7}
10	Seam-Sealed Gold-Plated Kovar Package	1.6×10^{-9}	5.4×10^{-9}

Sensors Nos. 6 and 8 therefore could not be recalibrated to provide the curves required to precisely interpret the sensor readings taken during the 50°C/60% RH exposure. However, their companion sensors (i.e., the other sensors used in the packages sealed with the same adhesives) were not damaged and, as shown in Figures 17 and 18, the initial calibration curves were reasonably the same (especially for Sensors Nos. 8 and 9). Therefore, the post-exposure calibration curve obtained for Sensor No. 7 was used to interpret the readings of both Sensors Nos. 6 and 7, and the post-exposure calibration curve obtained for Sensor No. 9 was used to interpret the readings of both Sensors Nos. 8 and 9. The post-exposure calibration curves for Sensors 7, 9, and 10 are given in Figures 19, 20 and 21, respectively. The initial calibration curves are also shown for comparison. Note that the shifts in the calibration curves for each of the three sensors are different. This was expected since the temperature exposures experienced by the three sensors were different (Table 7).

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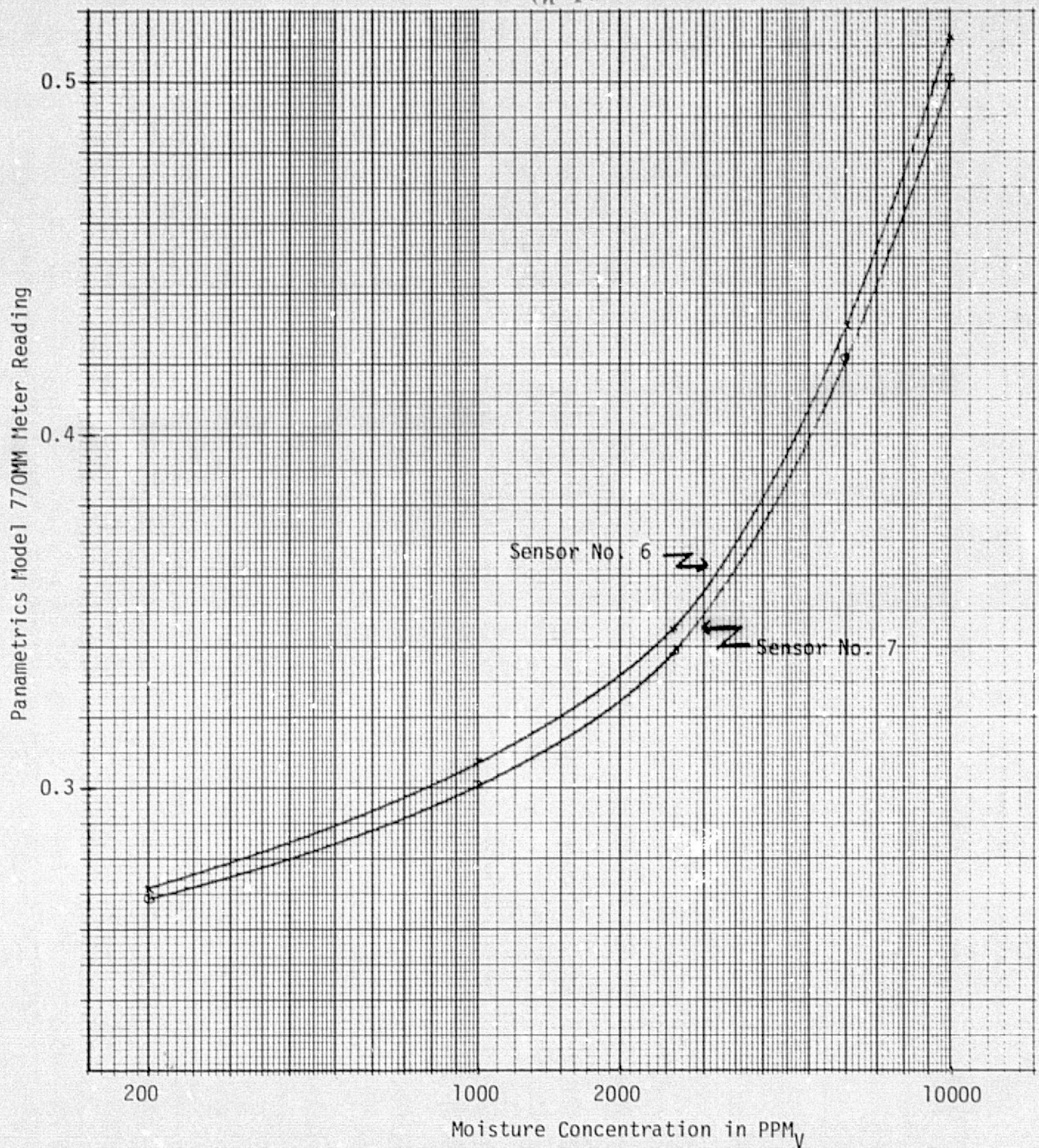


Figure 17. Initial Calibration Curves for Sensors Nos. 6 and 7 at 50°C

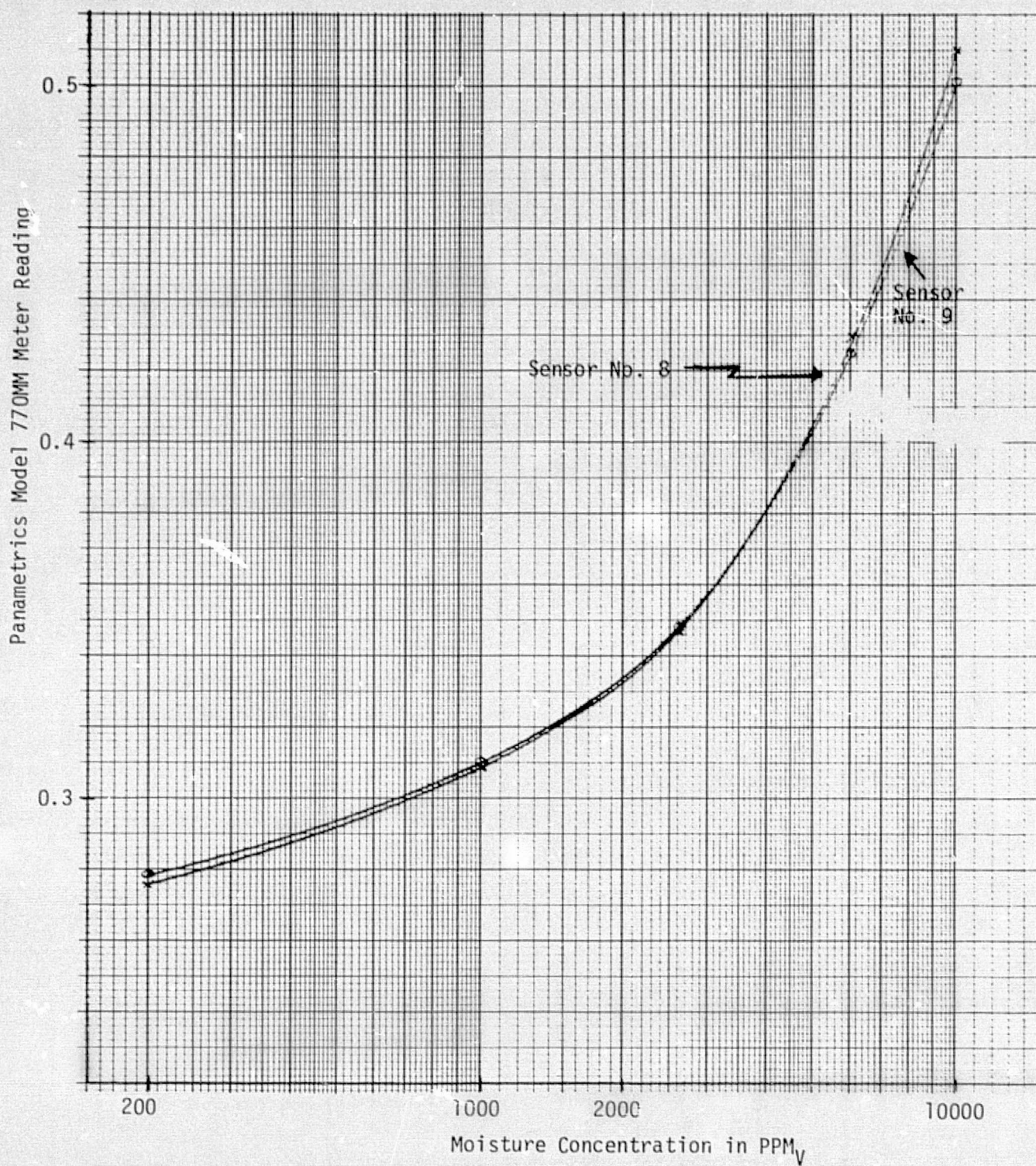


Figure 18. Initial Calibration Curves for Sensors No. 8 and 9 at 50°C

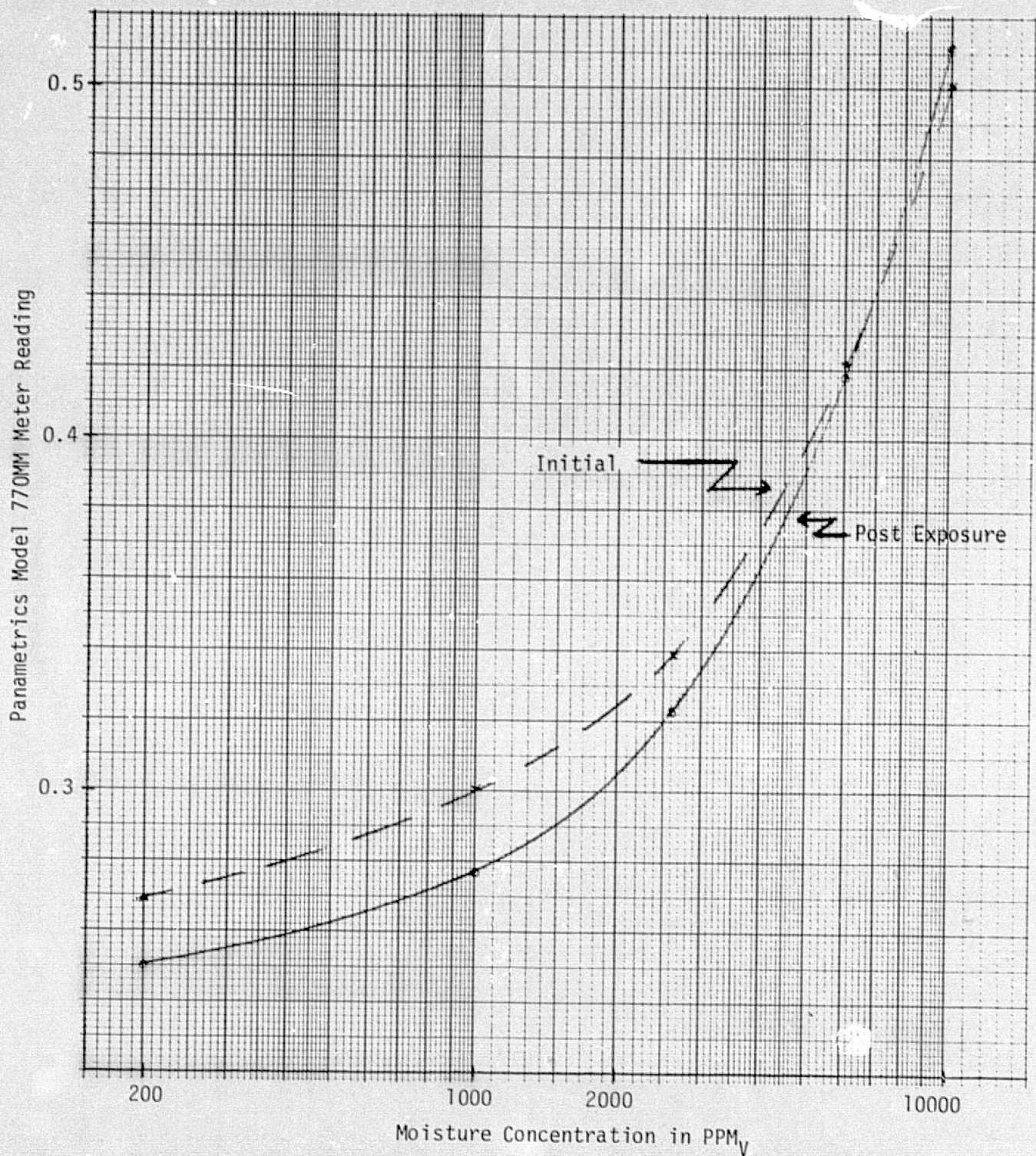


Figure 19. Initial and Post-Exposure (50°C/60% RH) Calibration Curves for Sensor No. 7 at 50°C

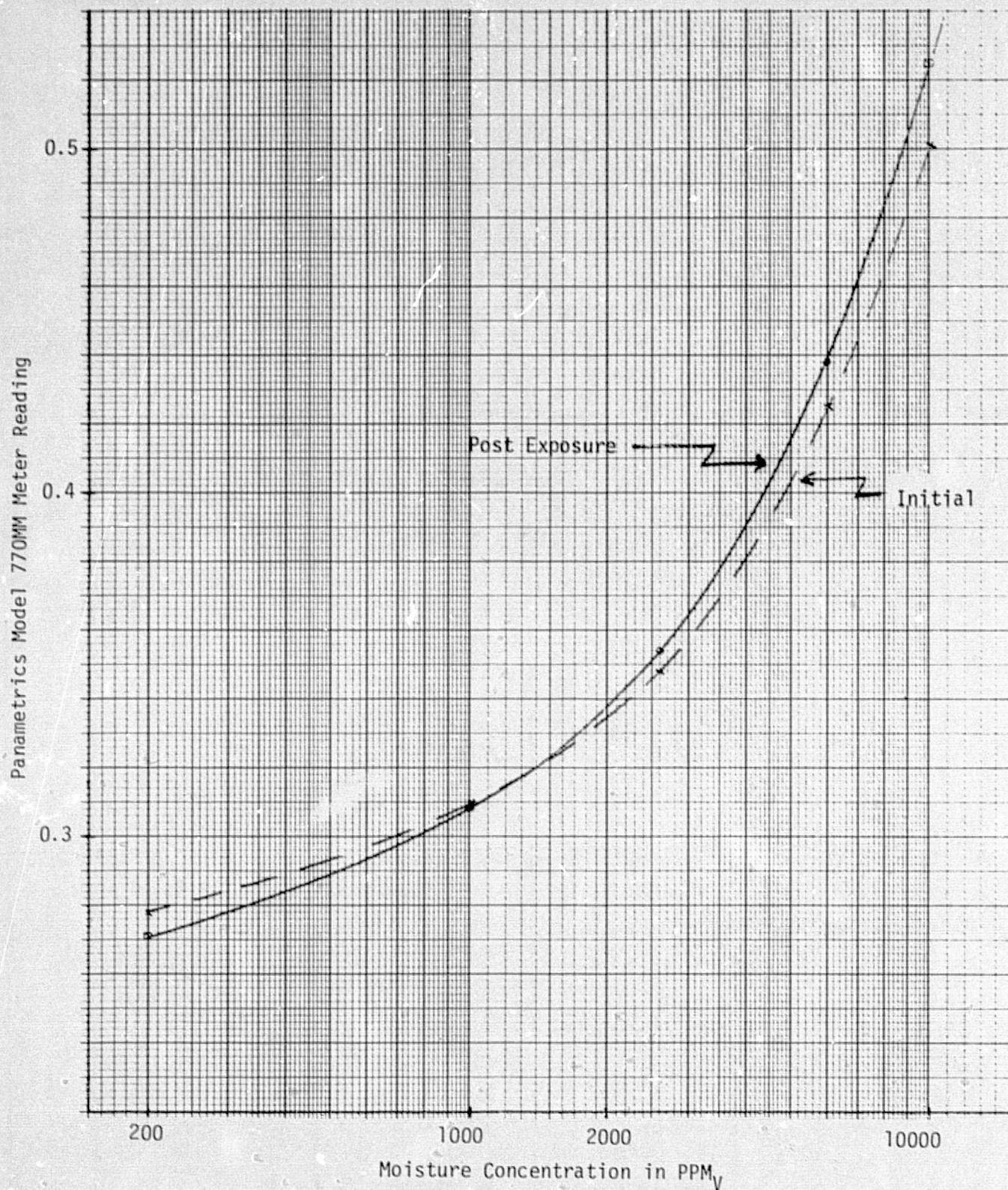


Figure 20. Initial and Post-Exposure (50°C/60% RH)
Calibration Curves for Sensor No. 9 at 50°C

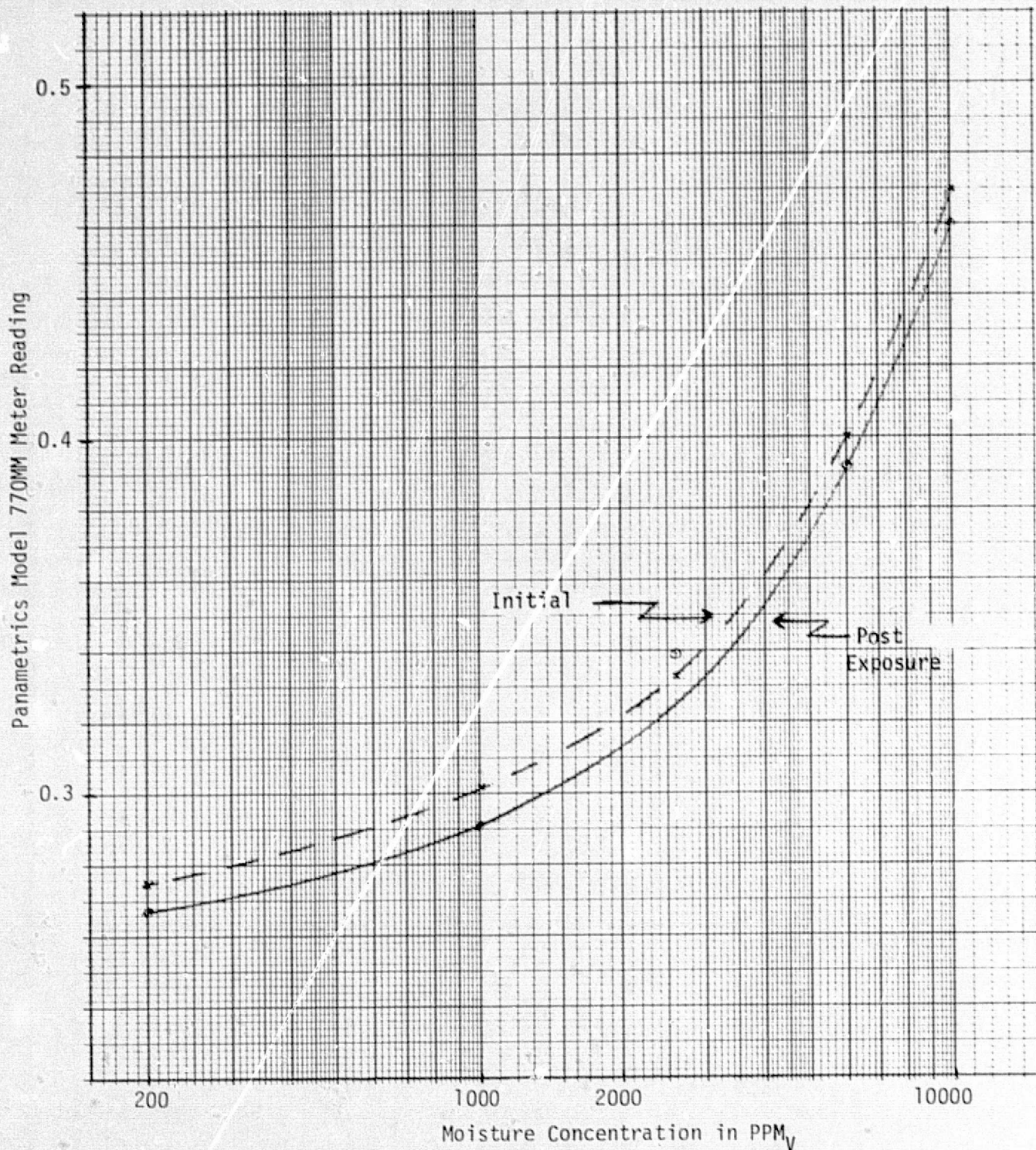


Figure 21. Initial and Post-Exposure (50°C/60% RH) Calibration Curves for Sensor No. 10 at 50°C

TABLE 7. TEMPERATURE EXPOSURES EXPERIENCED BY SENSORS

Sensor No.	Sealing Method	Temperature Exposure
6 & 7	Sealed With Ablebond 789-1 Epoxy	2 hours at 170°C plus 3 hours at 150°C
8 & 9	Sealed With Epo-Tek H77 Epoxy	1-1/2 hours at 150°C
10	Seam-sealed	Vacuum Baked for 4 hours at 135°C

Conversion of the sensor readings of Table 5 into moisture content in ppm_V using these post-exposure calibration curves is given in Table 8. Results are shown in graphical form in Figure 22. The results show that moisture permeated into all the packages (including the seam-sealed gold-plated Kovar package). The shapes of the curves indicate that moisture permeation into the seam-sealed gold-plated Kovar package was due to convection (Knudsen or Poiseuille flow) and that moisture permeation into the adhesive-sealed packages was due to diffusion (penetration through the lattice structure of the adhesives). Scaling from the graphs gives the following permeation rates:

- (a) 380 ppm_V/day for the seam-sealed gold-plated Kovar package
- (b) 150 to 215 ppm_V/day for the ceramic packages sealed with Ablebond 789-1
- (c) 1400 to 1750 ppm_V/day for the ceramic packages sealed with Epo-Tek H77.

The results obtained for the adhesive-sealed packages are of the order expected and verify the nonhermeticity of adhesive seals. The results also show that the quality of the seal obtained is dependent on the adhesive used. In the case of the two adhesives tested, Ablebond 789-1 was approximately an order of magnitude better than Epo-Tek H77 at the 50°C/60% RH exposure at which they were tested. The result obtained for the seam-sealed gold-plated Kovar package was surprising. Seal testing prior to and after the 50°C/60% RH exposure indicated leak rates of 1.6 and 5.4×10^{-9} atm cc/sec air equivalent, respectively, and the C_1 gross leak test indicated that the package was not a gross leaker. This is well within the seal test requirements of MIL-STD-883A. Possible explanations are that leak rates of even this small magnitude are

TABLE 8. MOISTURE CONTENT OF PACKAGES IN PPM_V DURING 50°C/60% RH EXPOSURE

Exposure (Days)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Gold-Plated Kovar Package Seam-Sealed
	Package No. 6 (Sensor No. 6)	Package No. 7 (Sensor No. 7)	Package No. 8 (Sensor No. 8)	Package No. 9 (Sensor No. 9)	Package No. 10 (Sensor No. 10)
0	< 100	< 100	< 100	< 100	110
1	< 100	< 100	< 100	< 100	225
2	< 100	< 100	100	< 100	410
3	< 100	< 100	540	315	680
4	< 100	< 100	1620	1020	1040
7	160	300	6400	4650	2250
8	230	460	8300	6150	2450
9	380	680	10,000	7700	2850
10	430	820	11,500	9000	3300
11	485	1060	12,900	10,300	3750
14	850	1740			4850
15	1030	1900			5300
16	1130	2170			5800
17	1300	2400			6000
18	1480	2540			6350
21	1900	3230			7450

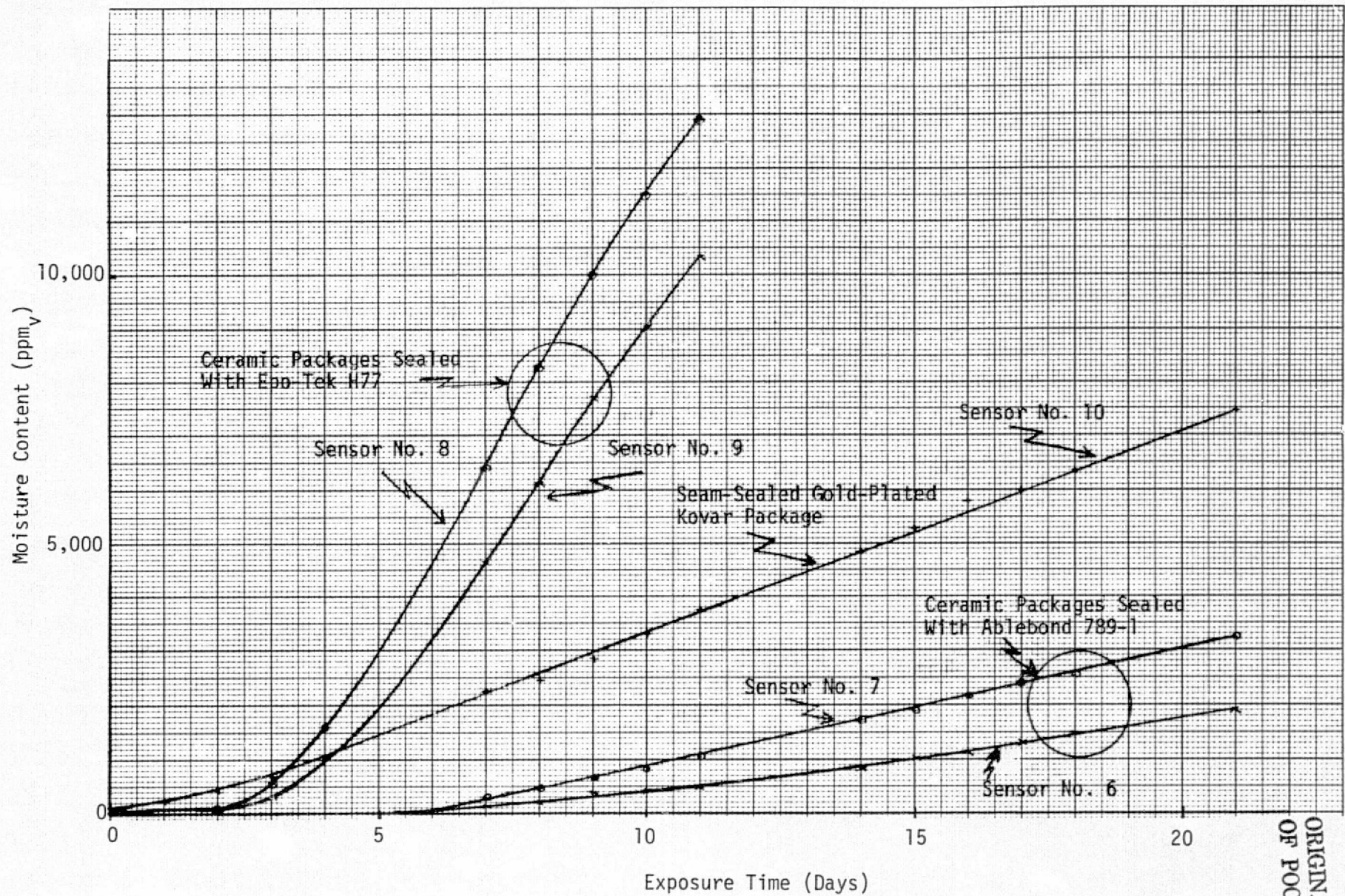


Figure 22. Moisture Permeation Into Packages Exposed to $50^\circ\text{C}/60\% \text{ RH}$

sufficient to permit moisture to enter the package, or that a leak developed at 50°C that was not detectable either at room temperature at which the fine leak test is performed or on brief exposure to 125°C as required in the C₁ gross leak test. In either case, this anomaly should be further investigated.

2.3.5 60°C/98% RH Exposure Results

The moisture sensor readings obtained during this exposure are given in Table 9. The moisture sensor in one of the ceramic packages sealed with Ablebond 789-1 (Sensor No. 11) behaved erratically within a few minutes after installation in the test chamber. Further testing of the sensor indicated that it had shorted. Subsequent leak testing indicated that the package had a fine leak rate of 3.2×10^{-7} atm cc/sec air equivalent (in excellent agreement with the previously measured value of 2.6×10^{-7} atm cc/sec air equivalent given in Table 10) and that it passed the C₁ gross leak test. Consequently, the observed behavior does not appear to be due to an inadequate package seal but to a faulty sensor. The other ceramic package sealed with Ablebond 789-1 (containing Sensor No. 12) and the seam-sealed gold-plated Kovar package (containing Sensor No. 15) were exposed for 15 days. However, exposure of the two ceramic packages sealed with Epo-Tek H77 (containing Sensors Nos. 13, and 14A) was terminated after five days because the sensor readings (based on the initial calibration curves) indicated that the moisture contents of these packages were in excess of 10,000 ppm_v.

The post-exposure calibration curves are given in Figures 23 through 27. The initial calibration curves are also shown for Sensors Nos. 11, 12, 13, and 15 for comparison. There is no initial calibration curve for Sensor No. 14A since the package containing this sensor was quickly prepared as a substitute for the package containing Sensor No. 14 when it was found that Sensor No. 14 was electrically open after package sealing. As can be seen, the shifts in the calibration curves are different. A difference in the shifts among the sensors exposed to the different sealing-preconditioning or curing time-temperature schedules, as listed in Table 11, is expected. However, reference to Figures 23 and 24 shows that there is a large difference in the shifts of the calibration curves for Sensors Nos. 11 and 12 which were exposed to essentially the same time-temperature conditions during sealing, but were not exposed to the 60°C/98% RH environment for the same length of time. Sensor

TABLE 9. MOISTURE SENSOR READINGS DURING 60°C/98% RH EXPOSURE

Exposure (Days)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Gold-Plated Kovar Package Seam-Sealed
	Sensor No. 11	Sensor No. 12	Sensor No. 13	Sensor No. 14A	Sensor No. 15
0	0.290	0.220	0.202	0.227	0.240
1	*	0.221	0.212	0.239	0.241
2		0.228	0.232	0.266	0.244
3		0.232	0.254	0.301	0.250
4		0.239	0.279	0.343	0.255
5		0.242	0.302	0.391	0.258
6		0.249			0.257
7		0.252			0.248
8		0.259			0.250
9		0.265			0.252
10		0.272			0.255
11		0.280			0.259
12		0.289			0.261
13		0.297			0.272
14		0.304			0.272
15		0.313			0.278

* Sensor behaved erratically within a few minutes after installation in test chamber. Further testing indicated sensor was shorted.

TABLE 10. INITIAL LEAK RATES OF PACKAGES EXPOSED TO 60°C/98% RH

Package Number	Description	Air Equivalent (atm cc/sec)
11	Ceramic Packages Sealed With Ablebond 789-1	2.6×10^{-7}
12		3.2×10^{-7}
13	Ceramic Packages Sealed With Epo-Tek H77	2.2×10^{-7}
14		2.0×10^{-7}
15	Seam-Sealed Gold-Plated Kovar Package	2.5×10^{-9}

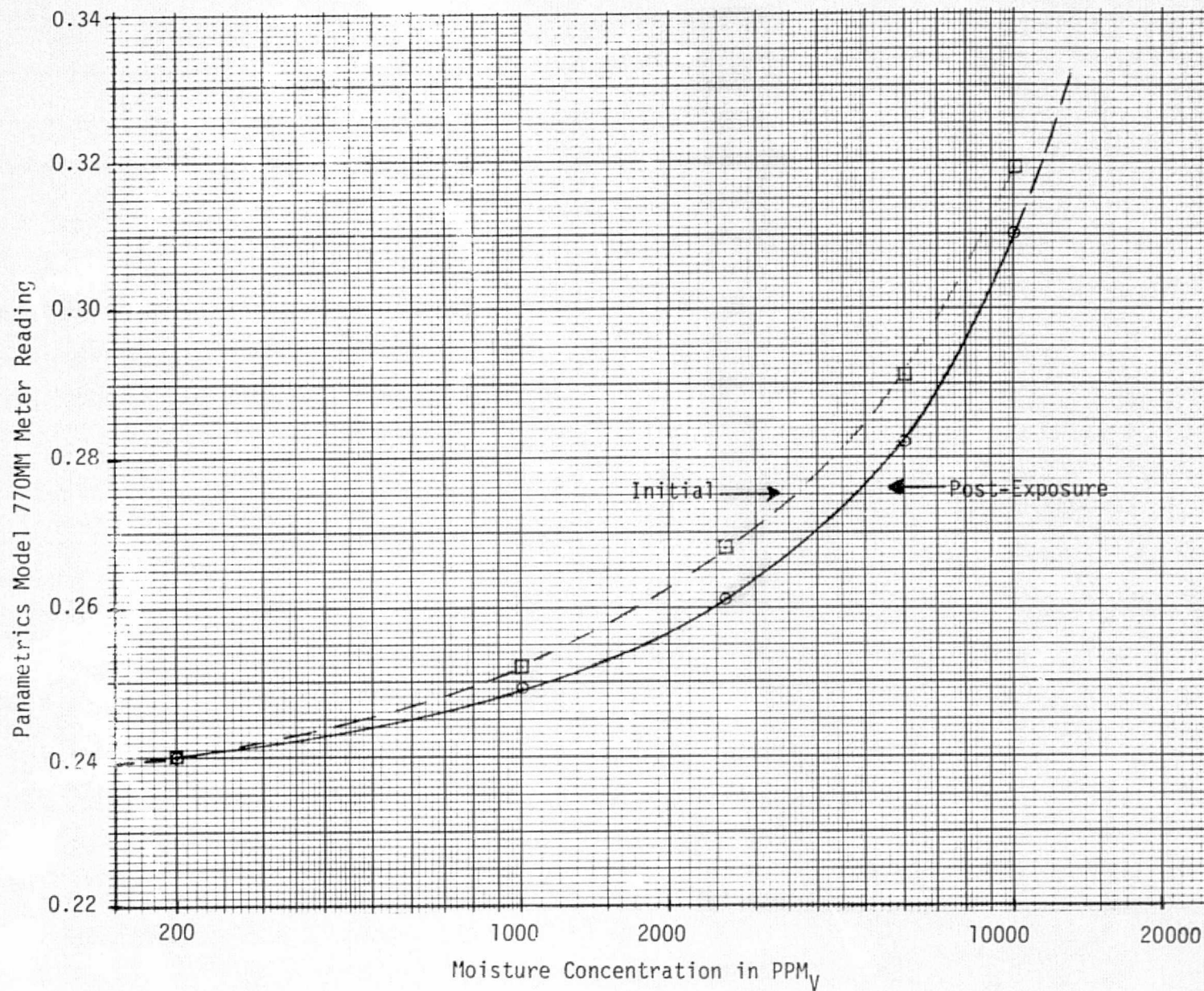


Figure 23. Initial and Post-Exposure (60°C/98% RH) Calibration Curves for Sensor No. 11 at 60°C

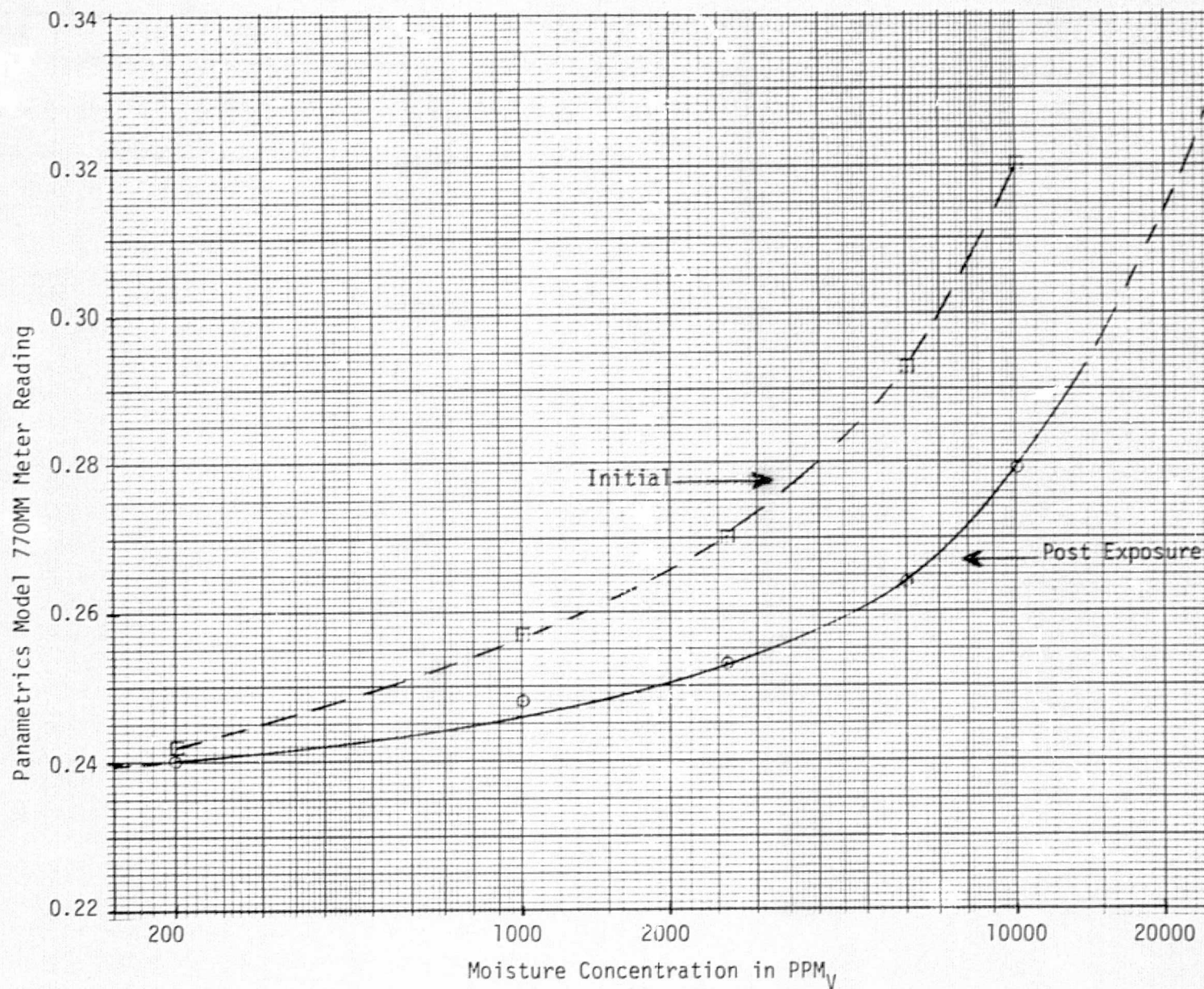


Figure 24. Initial and Post-Exposure ($60^\circ\text{C}/98\% \text{ RH}$) Calibration Curves for Sensor No. 12 at 60°C

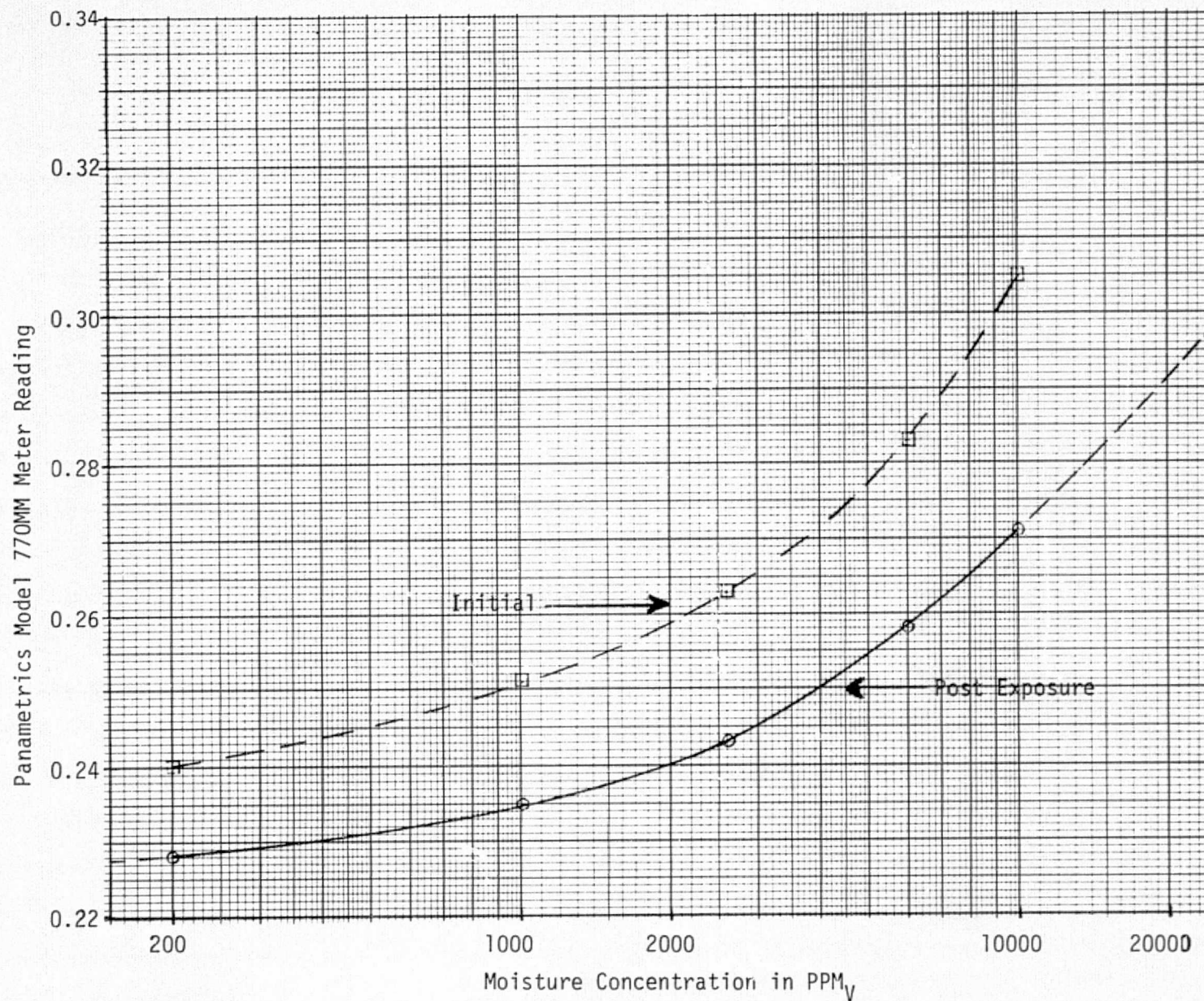


Figure 25. Initial and Post-Exposure (60°C/98% RH) Calibration Curves for Sensor No. 13 at 60°C

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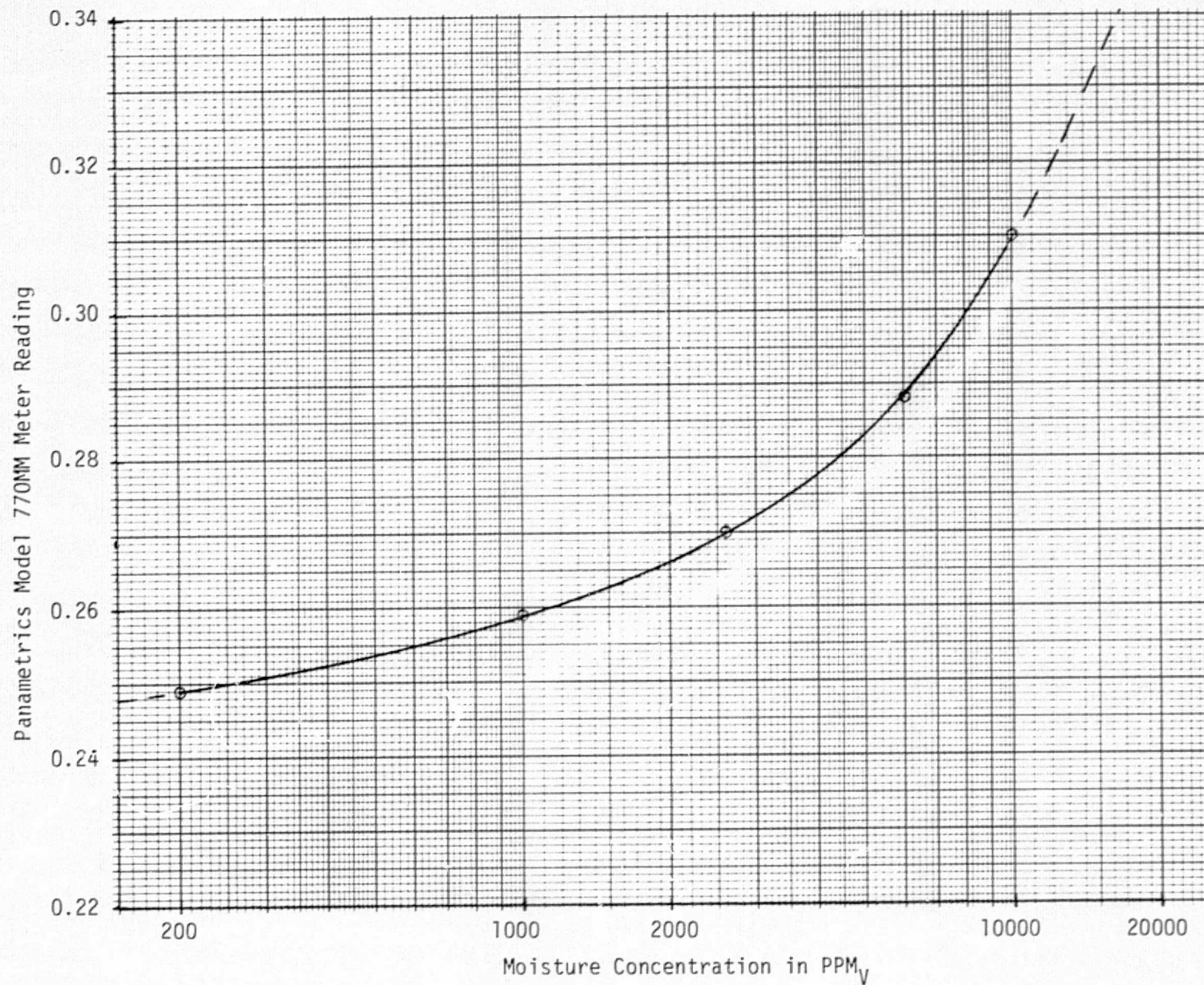


Figure 26. Post-Exposure (60°C/98% RH) Calibration Curve for Sensor No. 14A at 60°C

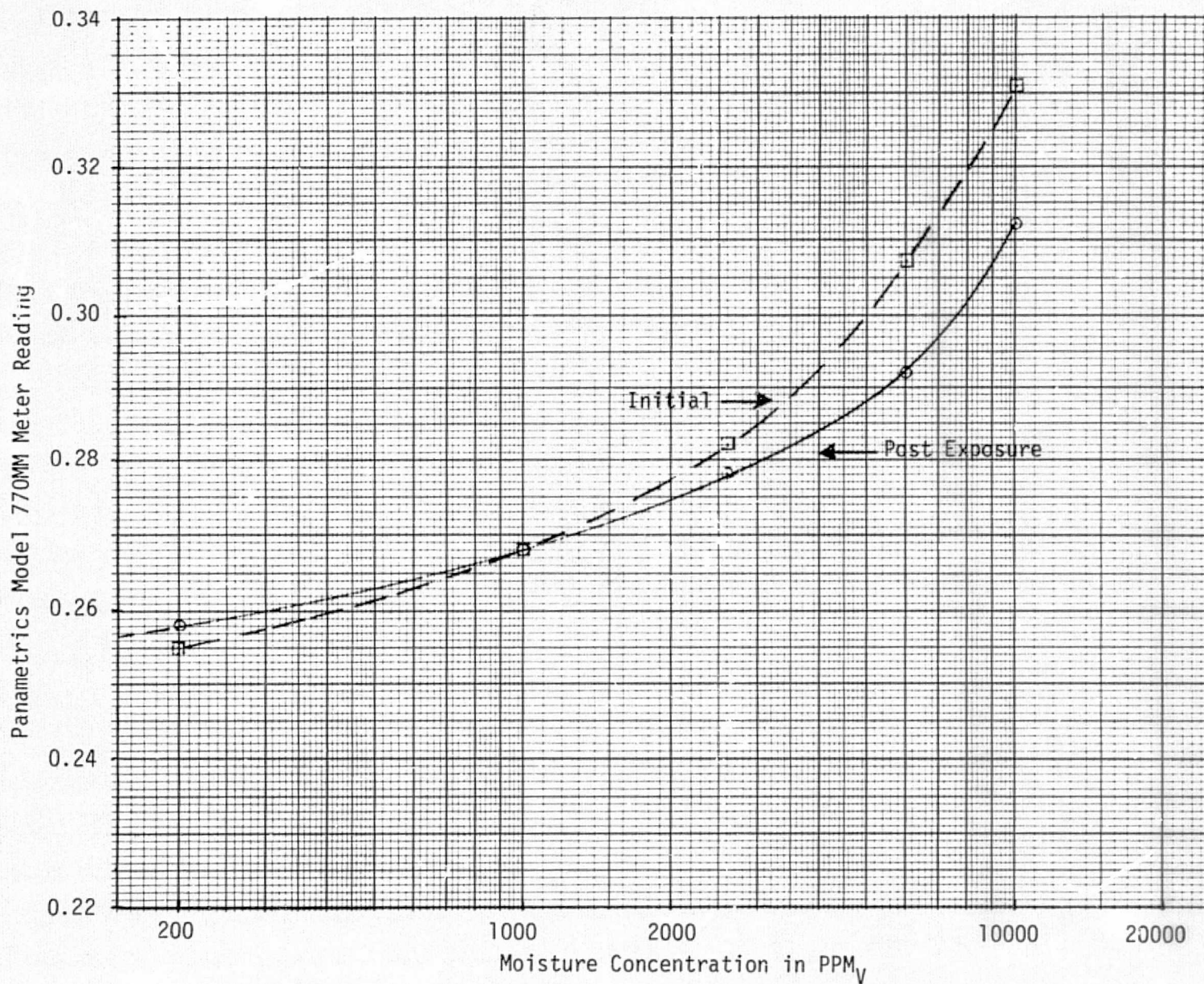


Figure 27. Initial and Post Exposure (60°C/98% RH) Calibration Curves for Sensor No. 15 at 60°C

TABLE 11. TEMPERATURE EXPOSURES EXPERIENCED BY SENSORS

Sensor No.	Sealing Method	Temperature Exposure
11	Sealed with Ablebond 789-1 Epoxy	2 Hrs at 170°C plus 1 Hr at 150°C
12		2 Hrs at 170°C plus 1-1/2 Hrs at 150°C
13 & 14A	Sealed with Epo-Tek H77 Epoxy	1-1/2 Hrs at 150°C
15	Seam-Sealed	Vacuum Baked for 4 Hrs at 135°C

No. 11 was exposed to this environment for only 30 minutes, while sensor No. 12 was exposed to it for 15 days. Thus, either the calibration shifts of the sensors vary from sensor to sensor, or the calibration continues to shift during the temperature-humidity exposure (in this case, 60°C/98% RH). Either condition is undesirable. The first requires that all sensors be recalibrated at the conclusion of the tests in which they have been used. The second invalidates their usefulness as a quantitative moisture monitor over an extended period of time since it implies that the only point for which an accurate value could be known is that taken just prior to the recalibration. Further work should be performed to resolve this situation.

Conversion of the sensor readings taken during the 60°C/98% RH exposure to moisture content in ppm_V using the post-exposure calibration curves is given in Table 12. Results are graphed in Figure 28. As in the case of the results for the 50°C/60% RH exposure, these results also showed that moisture permeated into all the packages (including the seam-sealed, gold-plated Kovar package). Also, as in the case of the 50°C/60% RH exposure, the shapes of the curves for the adhesive-sealed packages indicated that the permeation mechanism was diffusion (permeation through the lattice structure of the adhesives) rather than convection (permeation through channels, cracks, or flaws in the adhesives). The situation is not so clear for the seam-sealed gold-plated Kovar package. This package had an initial measured leak rate of 2.5×10^{-9} atm cc/sec air equivalent, and its moisture content increased very slowly to 100 ppm_V at the end of ten days exposure. However, after this, the moisture content increased very rapidly to 2600 ppm_V at the end of 15 days exposure. This could have been due to the fact that the package developed a larger leak due to the prolonged 60°C/98% RH exposure. Post-exposure leak tests were not performed to verify if this had occurred, but experience with the seam-sealed gold-plated Kovar package used in the 50°C/60% RH test indicates that measurement of the post-exposure leak rate probably would not have resolved this question anyway.

Scaling from the graphs of Figure 28 gives the following final permeation rates for the adhesive-sealed ceramic packages: (a) Ablebond 789-1: 2400 ppm_V/day , and (b) Epo-Tek H77: 14,000 ppm_V/day . These permeation rates are substantially larger than those obtained for the 50°C/60% RH exposure which were 150 to 215 ppm_V/day and 1400 to 1750 ppm_V/day for packages sealed with Ablebond 789-1 and Epo-Tek H77, respectively.

TABLE 12. MOISTURE CONTENT OF PACKAGES IN PPM_v DURING 60°C/98% RH EXPOSURE

Exposure (Days)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Seam-Sealed Gold- Plated Kovar Pkg.
	Package No. 11 (Sensor No. 11)	Package No. 12 (Sensor No. 12)	Package No. 13 (Sensor No. 13)	Package No. 14A (Sensor No. 14A)	Package No. 15 (Sensor No. 15)
0	7100	<100	<100	<100	<100
1	*	<100	<100	<100	<100
2		<100	600	2,000	<100
3		<100	4,850	8,300	<100
4		100	13,300	17,200	100
5		400	27,700	30,500	200
6		1,650			165
7		2,400			<100
8		4,400			<100
9		6,200			<100
10		8,050			100
11		10,200			250
12		12,600			360
13		14,900			1,550
14		17,000			1,550
15		19,800			2,600

*Sensor behaved erratically within a few minutes after installation in test chamber. Further testing indicated sensor was shorted, so no measurements were made.

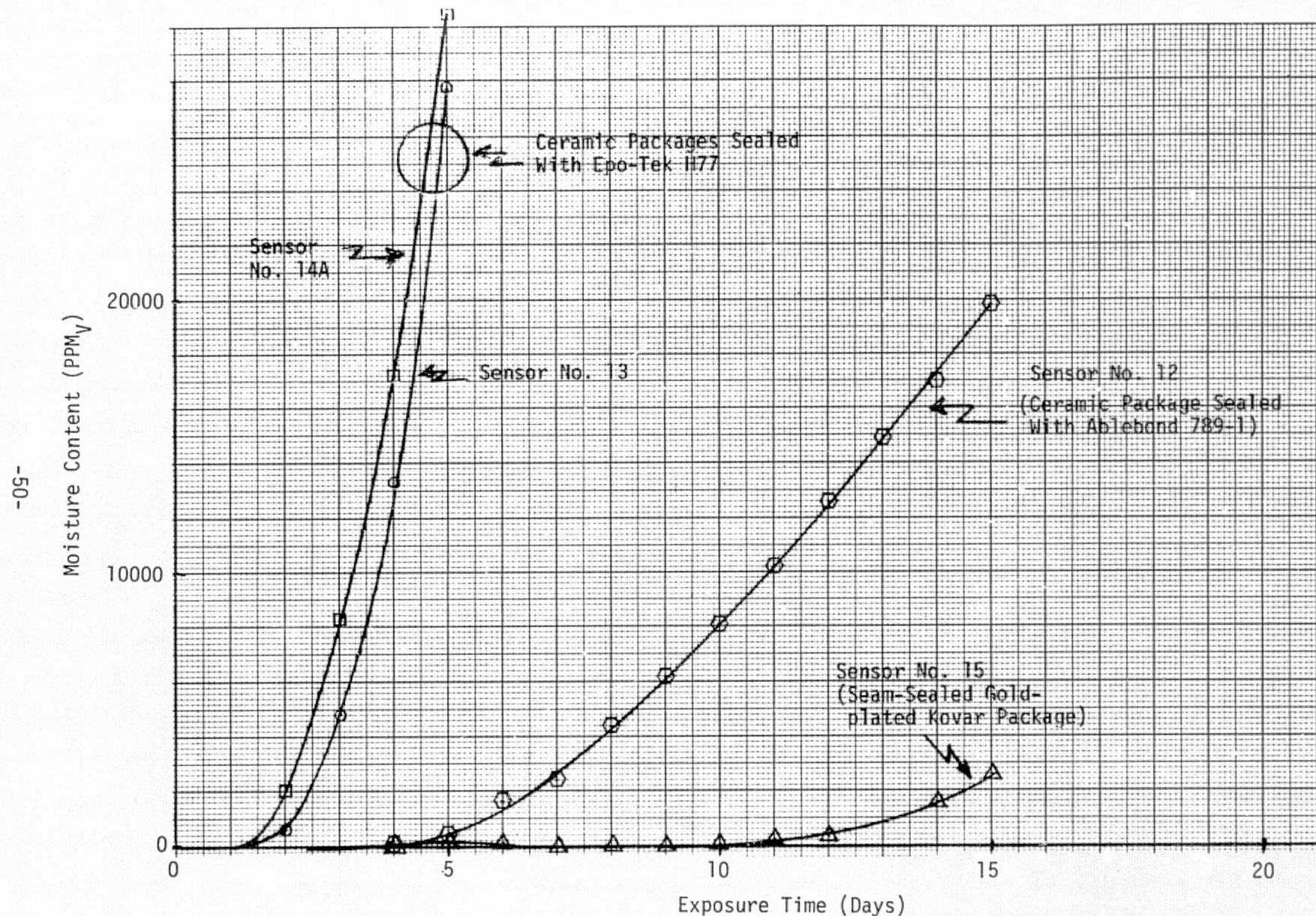


Figure 28. Moisture Permeation Into Packages Exposed to 60°C/98% RH

2.3.6 85°C/85% RH Exposure Results

The moisture sensor readings taken during this exposure are given in Table 13. It should be noted that after the packages were sealed, the sensor in one of the packages sealed with Epo-Tek H77 (Sensor No. 19) was found to be open. A similar situation occurred for the packages prepared for the 60°C/98% RH exposure (i.e.; after sealing, the sensor, Sensor No. 14, in one of the packages sealed with Epo-Tek H77 was found to be open). In both cases, further examination showed that the failures were not due to bad wire bonds but were sensor failures. It is not known whether it is simply coincidental that both failures observed occurred in Epo-Tek H77 adhesive-sealed packages or are due to contamination outgassed from this particular adhesive. Because of the high rate at which moisture permeated into the packages, the two ceramic packages sealed with Epo-Tek H77 (containing Sensors Nos. 18 and 19A) were removed after only 24 hours exposure and the test was terminated at the end of 52 hours. Post-exposure leak test results are given in Table 14 along with the leak rates measured prior to exposure. Comparison of the leak rates shows that the integrity of the packages was not affected by the 85°C/85% RH exposure. The post-exposure calibration curves are given in Figures 29 through 33. The initial calibration curves are also given for comparison. It should be noted that the shifts in the calibration curves are essentially the same for the two sensors (Sensors Nos. 16 and 17) exposed to the same curing time-temperature schedule (see Table 15).

Conversion of the sensor readings of Table 13 to moisture content in ppm_V based on the post-exposure calibration curves is given in Table 16. These results are graphed in Figure 34. As in the case of the previous exposures, these results showed that moisture permeated into all of the packages, including the seam-sealed gold-plated Kovar package. In this case, moisture permeation was very rapid and reached 6000 ppm_V in seven to ten hours for the ceramic packages sealed with Epo-Tek H77, 20 to 24 hours for the ceramic packages sealed with Ablebond 789-1, and approximately 30 hours for the seam-sealed gold-plated Kovar package. Calculation of final permeation rates for the packages sealed with Epo-Tek H77 would be meaningless. This probably also is the case for packages sealed with Ablebond 789-1 and for the seam-sealed gold-plated Kovar package. However, scaling from the graphs in these cases gives 24,000 to 84,000 ppm_V/day for packages sealed with Ablebond 789-1 and 9,600 ppm_V/day for the seam-sealed gold-plated Kovar package.

TABLE 13. MOISTURE SENSOR READINGS DURING 85°C/85% RH EXPOSURE

Exposure (Hours)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Gold-Plated Kovar Package Seam-Sealed
	Sensor No. 16	Sensor No. 17	Sensor No. 18	Sensor No. 19A	Sensor No. 20
0	0.228	0.230	0.222	0.268	0.239
1	0.230	0.230	0.240	0.289	0.240
2	0.231	0.230	0.241	0.289	0.242
4.5	0.236	0.232	0.252	0.291	0.250
5.5	0.238	0.235	0.259	0.292	0.251
7	0.240	0.238	0.268	0.297	0.255
8	0.241	0.239	0.275	0.301	0.258
24	0.260	0.258	0.438	0.482	0.275
28	0.262	0.259			0.278
32	0.269	0.262			0.280
48	0.289	0.282			0.295
52	0.293	0.290			0.298

TABLE 14. LEAK RATES OF PACKAGES EXPOSED TO 85°C/85% RH

Package Number	Description	Air Equivalent (atm cc/sec)	
		Prior to Exposure	After Exposure
16	Ceramic Packages Sealed With Ablebond 789-1	2.7×10^{-7}	1.6×10^{-7}
17		2.3×10^{-7}	2.4×10^{-7}
18	Ceramic Packages Sealed With Epo-Tek H77	1.4×10^{-7}	3.0×10^{-7}
19A		9.8×10^{-8}	1.3×10^{-7}
20	Seam-Sealed Gold-Plated Kovar Package	2.1×10^{-9}	2.6×10^{-9}

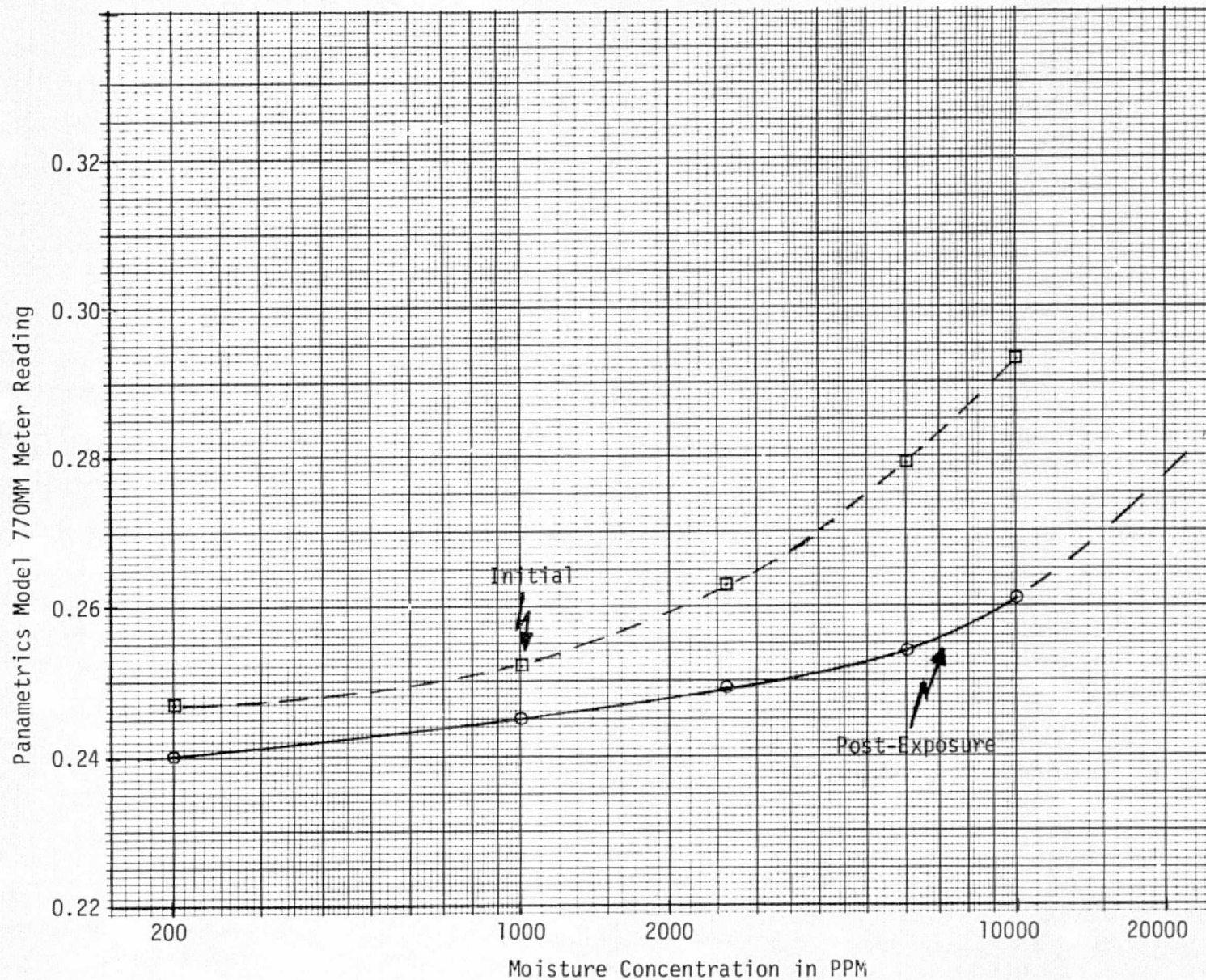


Figure 29. Initial and Post-Exposure (85°C/85% RH) Calibration Curves for Sensor No. 16 at 85°C

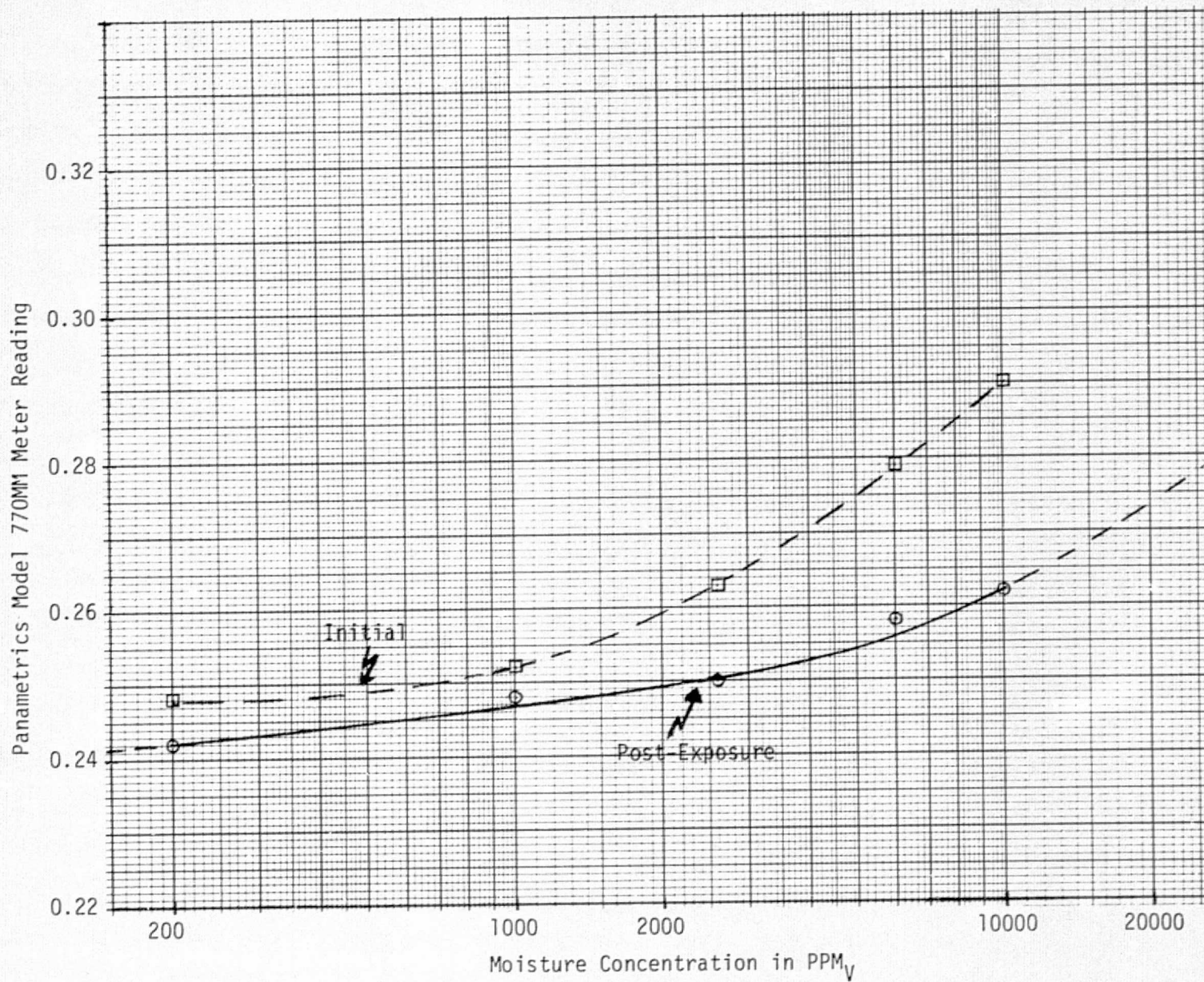


Figure 30. Initial and Post-Exposure (85°C/85% RH) Calibration Curves for Sensor No. 17 at 85°C

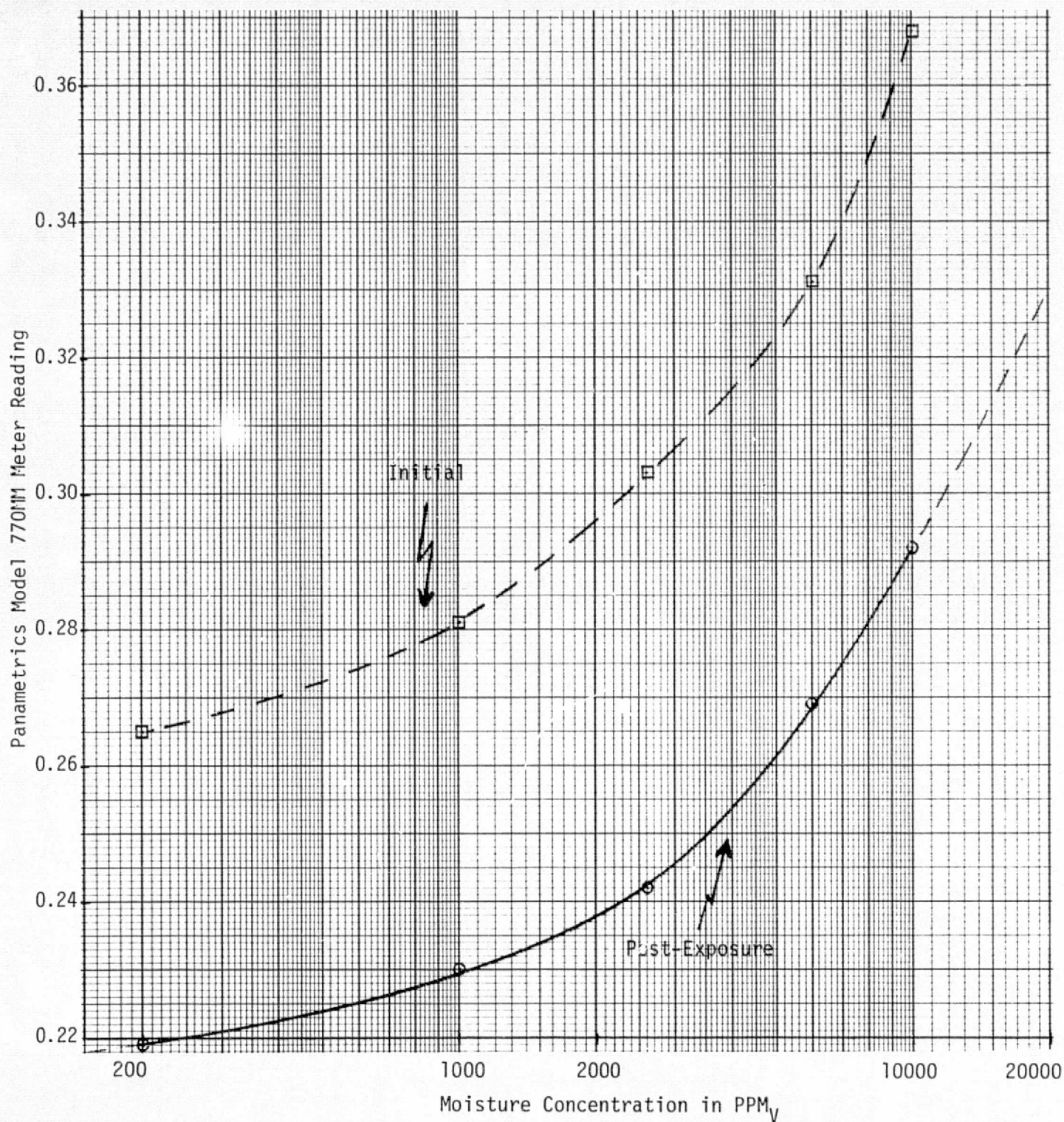


Figure 31. Initial and Post-Exposure (85°C/85% RH) Calibration Curves for Sensor No. 18 at 85°C

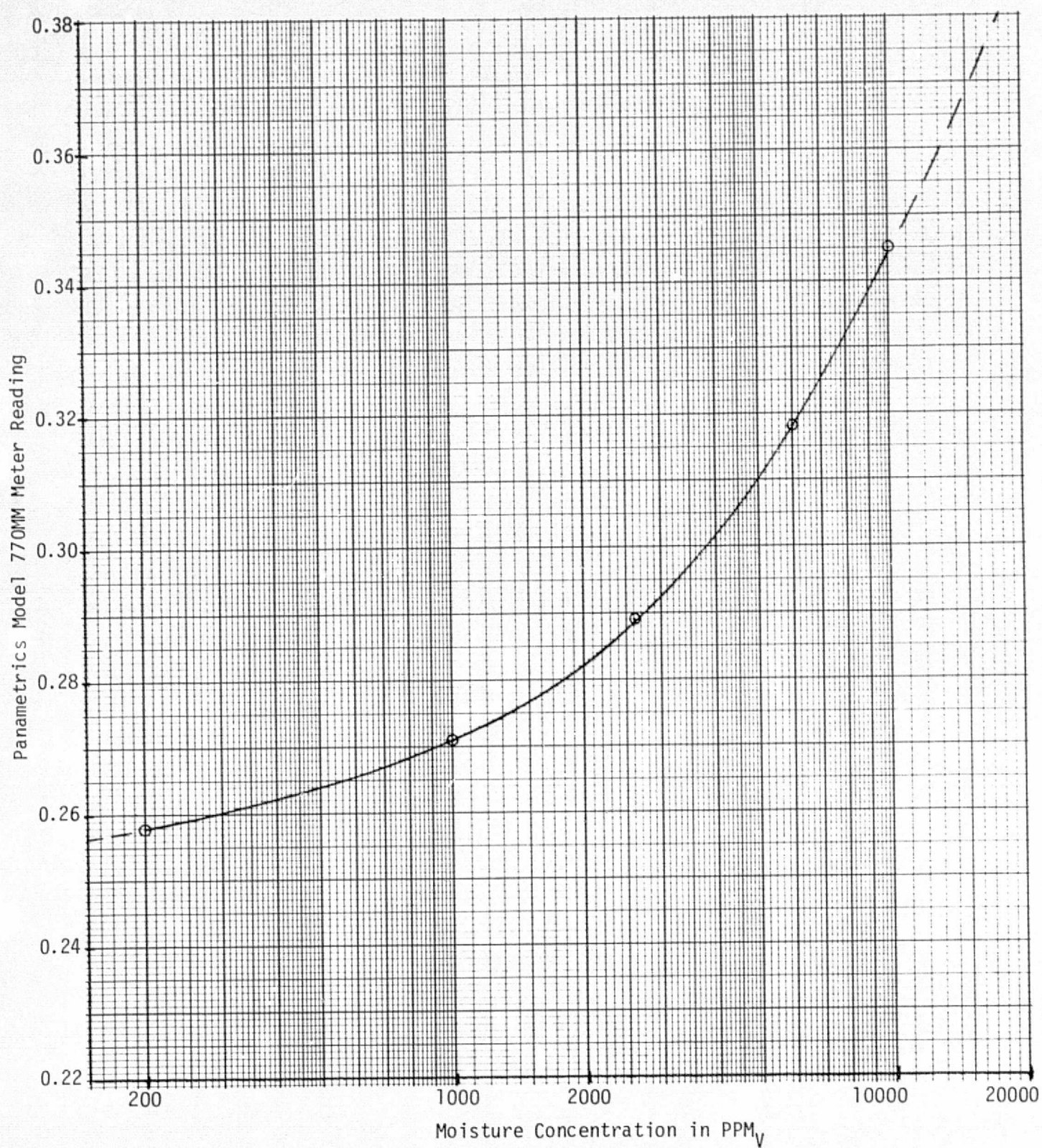


Figure 32. Post-Exposure (85°C/85% RH) Calibration Curve for Sensor No. 19A at 85°C

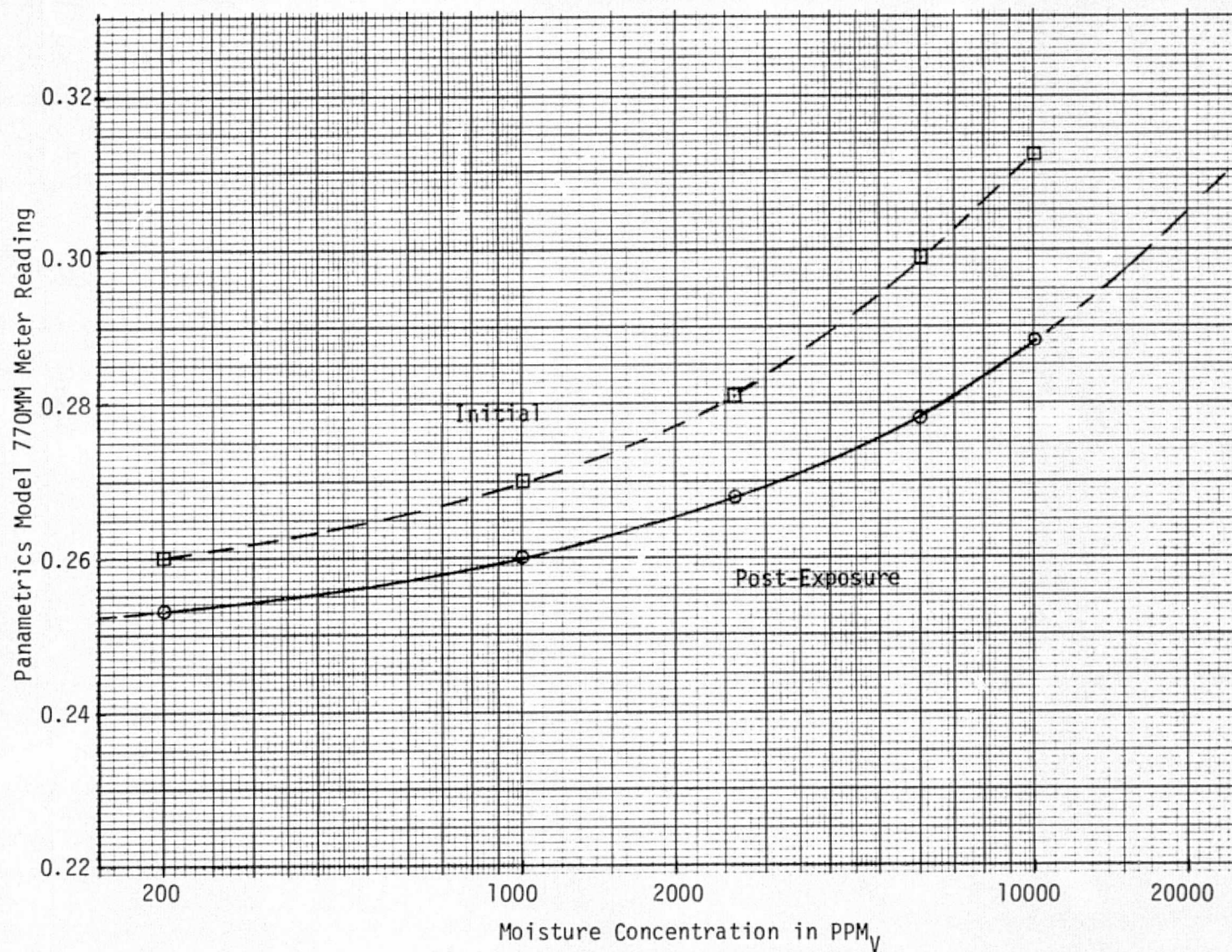


Figure 33. Initial and Post-Exposure (85°C/85% RH) Calibration Curves for Sensor No. 20 at 85°C

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TABLE 15. TEMPERATURE EXPOSURES EXPERIENCED BY SENSORS

Sensor No.	Sealing Method	Temperature Exposure
16 & 17	Sealed With Ablebond 789-1 Epoxy	2 hours at 170°C plus 1 hour at 150°C
18 & 19	Sealed With Epo-Tek H77 Epoxy	1-1/2 hours at 150°C
20	Seam-Sealed	Vacuum Baked for 4 hours at 135°C

TABLE 16. MOISTURE CONTENT OF PACKAGES IN PPM_v DURING 85°C/85% RH EXPOSURE

Exposure (Hours)	Ceramic Packages Sealed With Ablebond 789-1		Ceramic Packages Sealed With Epo-Tek H77		Gold-Plated Kovar Package Seam-Sealed
	Package No. 16 (Sensor No. 16)	Package No. 17 (Sensor No. 17)	Package No. 18 (Sensor No. 18)	Package No. 19A (Sensor No. 19A)	Package No. 20 (Sensor No. 20)
0	<100	<100	370	770	<100
1	<100	<100	2,300	2,600	<100
2	<100	<100	2,400	2,600	<100
4.5	<100	<100	3,800	2,800	<100
5.5	~100	<100	4,700	2,930	~100
7	200	<100	5,900	3,480	370
8	270	<100	7,000	3,900	740
24	9,500	7,300	94,000	72,000	4,800
28	10,500	8,000			6,000
32	14,500	10,000			6,700
48	29,500	31,000			13,700
52	33,500	45,000			15,300

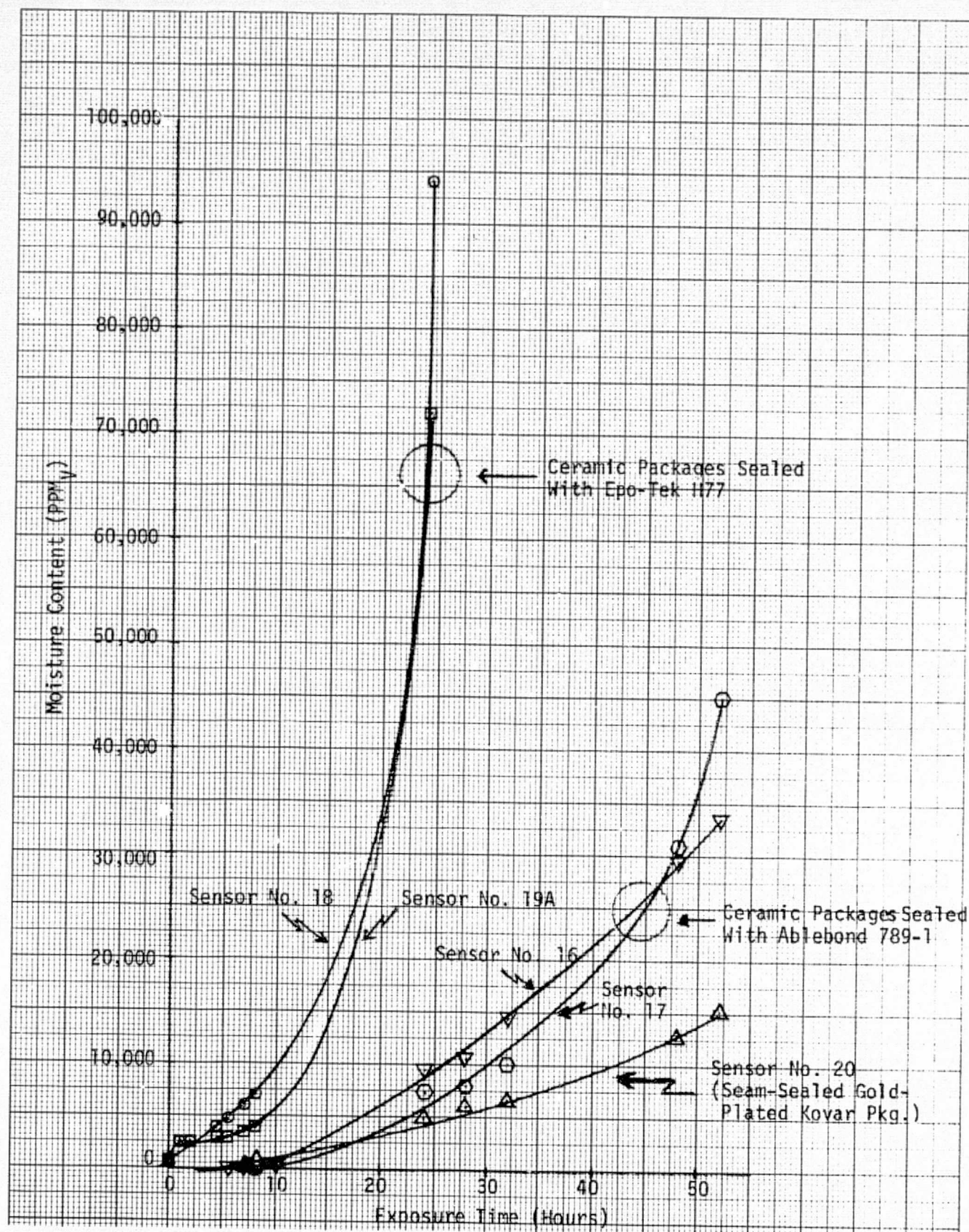


Figure 34. Moisture Permeation Into Packages Exposed to 85°C/85% RH

2.4 DISCUSSION OF RESULTS AND CONCLUSIONS

2.4.1 Comparison of Results Obtained at the Different Temperature-Humidity Exposures

To simplify comparison, the results obtained at the four different temperature-humidity environments have been replotted in Figures 35, 36 and 37 for each of the three different package types (Ablebond 789-1 adhesive-sealed ceramic packages, Epo-Tek H77 adhesive-sealed ceramic packages, and seam-sealed gold-plated Kovar packages). Before commenting on these results, it should be noted that the four exposure environments of 25°C/98% RH, 50°C/60% RH, 60°C/98% RH, and 85°C/85% RH are increasingly severe; not only because their temperatures are higher, but also because their moisture vapor pressures or moisture contents are higher (see Table 17).

2.4.1.1 Adhesive-Sealed Ceramic Packages

Review of Figures 35 and 36 shows that the permeation of moisture into the adhesive-sealed ceramic packages behaved as expected (i.e.; became increasingly more rapid at the increasingly severe temperature-humidity exposures). It is also evident from these Figures that the moisture permeation rates at the successively more severe exposures increased by much larger factors than those by which the moisture vapor pressures for these exposures increased (i.e.; 2.38, 2.64 and 2.52). This is also as expected due to the exponential effect of temperature on the permeation rate as shown below. Simplifying the expression for the permeation rate due to diffusion as derived from Fick's law and expressed in the Appendix of Reference 2 gives the following:

$$J = \frac{A}{d} K_0 e^{-E/RT} \Delta p$$

In this expression, for the present case, A is the surface area and d is the width of the adhesive seal, Δp is the moisture vapor differential pressure across the adhesive seal, and $K_0 e^{-E/RT}$ is the permeation coefficient or permeability of the adhesive. In the expression, for the permeability, R is the gas constant, T is the exposure temperature, $K_0 = D_0 S_0$, and $E = E' + E''$ where D_0 is the diffusion constant, S_0 is the solubility constant, and E' and E'' are the activation energies for the diffusion and solution processes, respectively.

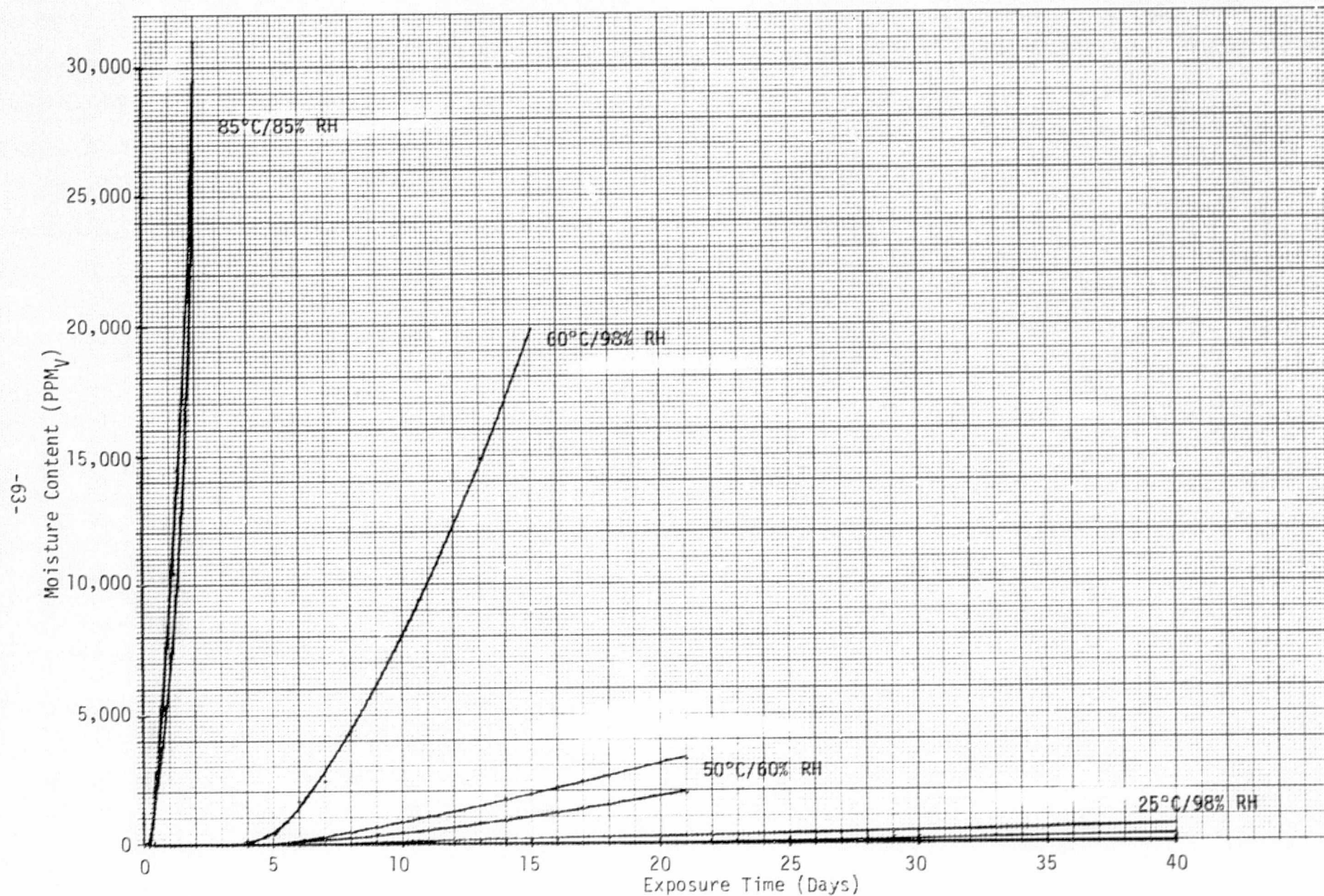


Figure 35. Comparison of Moisture Permeation Into Ablebond 789-1 Adhesive-Sealed Ceramic Packages at Different Temperature-Humidity Exposures

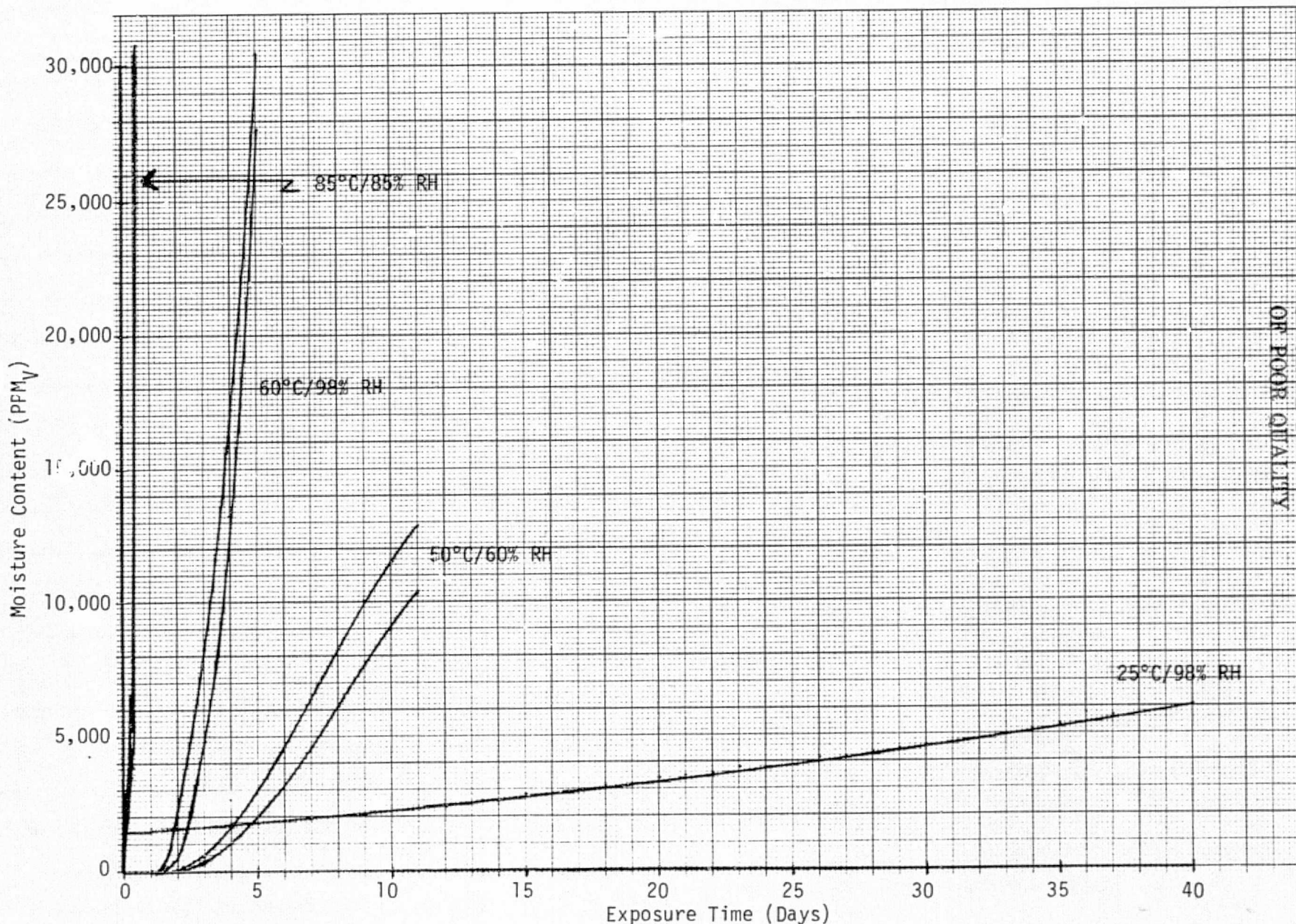


Figure 36. Comparison of Moisture Permeation Into Epo-Tek H77 Adhesive-Sealed Ceramic Packages at Different Temperature-Humidity Exposures

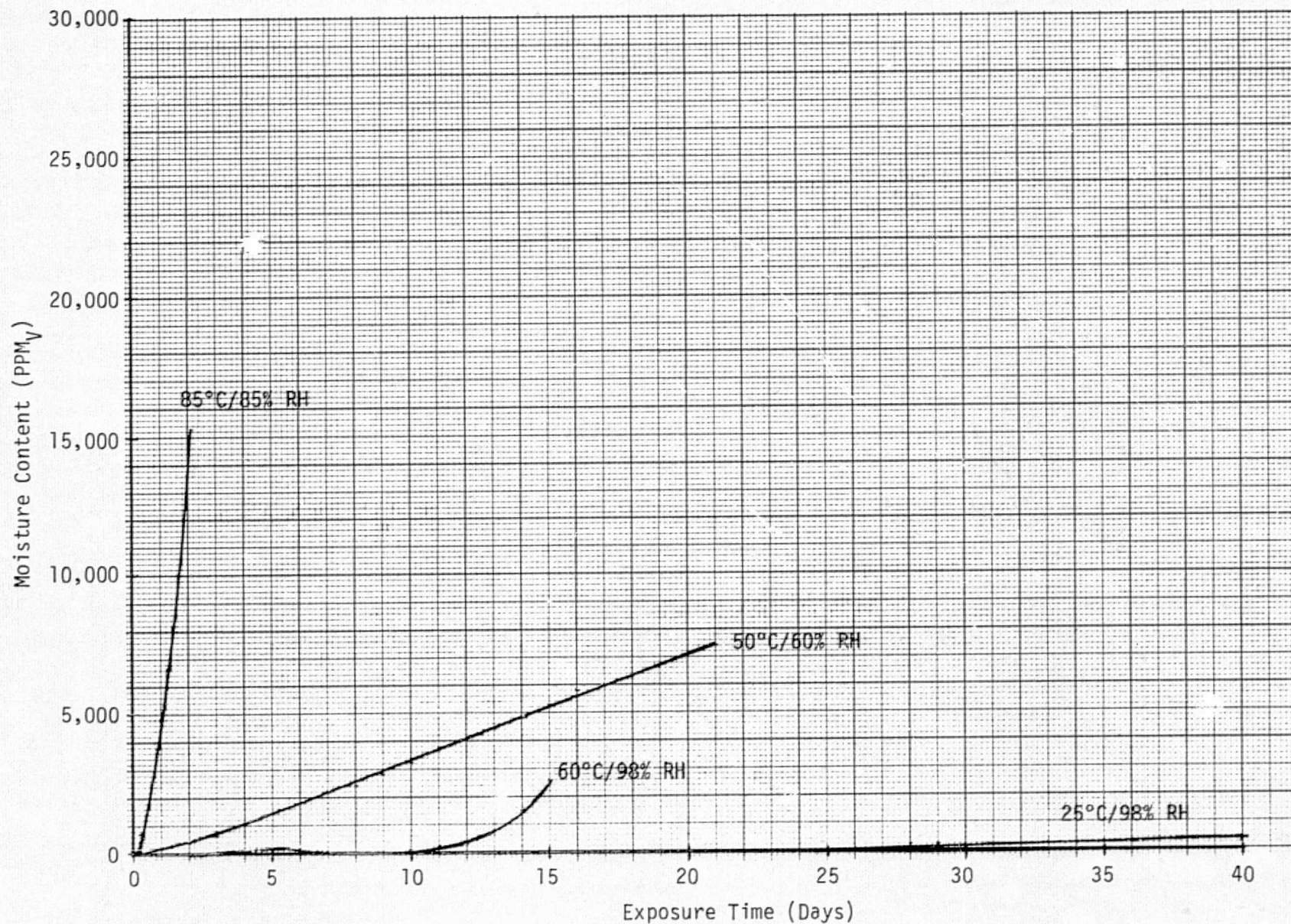


Figure 37. Comparison of Moisture Permeation Into Seam-Sealed Gold-Plated Kovar Packages at Different Temperature-Humidity Exposures

TABLE 17. MOISTURE VAPOR PRESSURES AND MOISTURE CONTENTS OF THE FOUR EXPOSURE ENVIRONMENTS

Exposure Environment	Moisture Vapor Pressure (Torr)	Moisture Content (ppm _v)
25°C/98% RH	23.28	30,600
50°C/60% RH	55.51	73,000
60°C/98% RH	146.39	192,600
85°C/85% RH	368.56	484,900

The final permeation rates for the adhesive-sealed ceramic packages at the different exposure environments are summarized in Table 18. These values were scaled directly from the graphs of the experimental data. As these results indicate, Ablebond 789-1 is approximately an order of magnitude better than Epo-Tek H77. The times required at the different exposure environments for the moisture contents of the adhesive-sealed ceramic packages to reach 6000 ppm_v (the maximum moisture content presently allowed by MIL-STD-883B for hybrid packages) are given in Table 19. Collectively, the results of Tables 18 and 19 show that the tested adhesives, Ablebond 789-1 and Epo-Tek H77, do not result in package seals that can meet the moisture content requirement of MIL-STD-883B even when exposed to the relatively benign environment of 25°C/98% RH. As a final point in this discussion, it is interesting to note that the result of 9 days obtained in this study for the moisture content of the Ablebond 789-1 adhesive-sealed package to reach 6000 ppm_v when exposed to a 60°C/98% RH environment compares closely with the 5.5 to 8 days previously obtained in the Phase II study for similarly sealed ceramic packages exposed to the same environment.

2.4.1.2 Seam-Sealed Gold-Plated Kovar Packages

The results shown in Figure 37 for the seam-sealed (brazed using gold-tin preforms) gold-plated Kovar packages were unexpected. These results show that moisture permeated all of these packages also. All packages were fine and gross-leak tested per MIL-STD-883A, Method 1014.1, Test Conditions A₂ and C₁ prior to exposure and all packages except the one exposed to the 60°C/98% RH environment were fine and gross-leak tested after exposure. In all cases, the measured fine leak rates were in the low-to-mid 10⁻⁹ atm cc/sec air equivalent region as shown in Table 20, and all packages passed the gross leak test. The final moisture permeation rates scaled from the graphs were as follows:

25°C/98% RH	21 ppm _v /day
50°C/60% RH	380 ppm _v /day
60°C/98% RH	1,050 ppm _v /day
85°C/85% RH	9,600 ppm _v /day

TABLE 18. FINAL PERMEATION RATES IN PPM_V/DAY FOR ADHESIVE-SEALED CERAMIC PACKAGES AT THE DIFFERENT TEMPERATURE-HUMIDITY EXPOSURES

Exposure Environment	Ablebond 789-1 Adhesive-Sealed Ceramic Packages	Epo-Tek H77 Adhesive-Sealed Ceramic Packages
25°C/98% RH	19 to 26	146
50°C/60% RH	150 to 215	1400 to 1750
60°C/98% RH	2400	14,000
85°C/85% RH	24,000 to 84,000	Too large to measure

TABLE 19. TIME IN DAYS FOR INTERNAL MOISTURE CONTENT OF ADHESIVE-SEALED CERAMIC PACKAGES TO REACH 6000 PPM_V

Exposure Environment	Ablebond 789-1 Adhesive-Sealed Ceramic Packages	Epo-Tek H77 Adhesive-Sealed Ceramic Packages
25°C/98% RH	236 to 332	40
50°C/60% RH	34 to 48	7 to 8
60°C/98% RH	9	≈ 3
85°C/85% RH	~ 1	< 0.5

TABLE 20. LEAK RATES OF SEAM-SEALED GOLD-PLATED KOVAR PACKAGES

Exposure Environment	Air Equivalent Leak Rates (atm cc/sec)	
	Prior to Exposure	After Exposure
25°C/98% RH	2.4×10^{-9}	3.4×10^{-9}
50°C/60% RH	1.6×10^{-9}	5.4×10^{-9}
60°C/98% RH	2.5×10^{-9}	Not measured
85°C/85% RH	2.1×10^{-9}	2.6×10^{-9}

The times required for the moisture contents of these packages to reach 6000 ppm_v (either calculated from the above data or scaled directly from the graphs) were:

25°C/98% RH	304 days
50°C/60% RH	17 days
60°C/98% RH	18 days
85°C/85% RH	1.25 days

Possible explanations are that leak rates of even this small magnitude, which are well within the requirements of MIL-STD-883A, are sufficient to permit moisture to enter the packages at elevated temperatures; or that leaks develop at the elevated temperatures that are not detectable either at room temperature at which the fine leak test is performed or on brief exposure to 125°C as required in the C₁ gross leak test. It is recommended that tests be performed to evaluate the validity of performing the fine leak test per Test Condition A₂ at room temperature. Such tests should consist of making fine leak rate measurements on the same packages at several different temperatures spanning the expected exposure range, as well as at room temperature, and comparing the results to determine if the measured leak rates are temperature dependent.

2.4.2 Repeatability of Moisture Sensor Calibration

Testing to check the repeatability of the calibration curves obtained for the Mini-Mod-A moisture sensors showed that while the repeatability was not excellent, it was at least reasonably good. Results for the worst case indicated repeatability variations of +12.5, +16.9, +6.6, and +5.6% at approximately 250, 1000, 5000, and 9000 ppm_v moisture values. This is well within the +25% accuracy of the moisture sensors specified by the manufacturer.

2.4.3 Changes in Moisture Sensor Calibration After 150°C Exposure

As discussed in Section 2.1.1, it is stated in the vendor's brochure that the Mini-Mod-A may be exposed to 150°C for two hours in dry air to cure the adhesive used to mount the sensor and baked at 150°C in vacuum for up to 30 hours. This implies that such exposures will not significantly shift the

calibration curve and that interpretation of measurements using the calibration curve supplied with the sensor will be accurate to within $\pm 25\%$. Data at four selected moisture values (200, 1000, 5000 and 8000 ppm_v) for the initial calibration curves are compared with those from the post-exposure calibration curves for five sensors exposed to cure schedules of 1.5 hours at 150°C (see Table 21). Post-exposure data within the $\pm 25\%$ tolerance range of the initial data are indicated in brackets. Only two moisture values for one sensor and three for another are within the $\pm 25\%$ tolerance range. Sensors Nos. 3 and 4 were exposed to the 25°C/98% RH environment, Sensor No. 9 to the 50°C/60% RH environment, Sensor No. 13 to the 60°C/98% RH environment, and Sensor No. 18 to the 85°C/85% RH environment. On the basis of these results, it appears that the calibration of the sensors changes during the temperature-humidity exposures. The very large shifts of Sensors Nos. 13 and 18, compared to the smaller shifts of Sensors Nos. 3, 4 and 9, seem to substantiate this conclusion.

TABLE 21. COMPARISON OF MOISTURE VALUES SCALED FROM
INITIAL AND POST-EXPOSURE CALIBRATION CURVES

Moisture Value Initial Calibration Curve (ppm _v)	Allowed Range (+25%) (ppm _v)	Moisture Value Post-Exposure Calibration Curve (ppm _v)				
		Sensor No. 3 Figure 13	Sensor No. 4 Figure 14	Sensor No. 9 Figure 20	Sensor No. 13 Figure 25	Sensor No. 18 Figure 31
200	150 to 250	420	600	300	2000	5500
1000	750 to 1250	1300	1600	[1000]	4300	8000
5000	3750 to 6250	[6000]	6600	[4500]	12400	17800
8000	6000 to 10000	[9300]	10500	[7100]	21800	?

3.0 SUMMARY

This study was a supplement to the Phase II Study with the objective of further investigating the susceptibility of adhesive-sealed ceramic packages to moisture permeation. In this study the two adhesives, Ablebond 789-1 and Epo-Tek H77, were evaluated as package sealants. These adhesives were previously selected during the Phase II Study as the most promising candidates for this application from a group of ten adhesives. Their selection was based on their performance (i.e.; their ability to seal packages that retained their seal integrity) after individual exposures to four increasingly severe temperature-humidity environments and both individual and sequential exposures to the Class A test environments specified in Method 5004.2 of MIL-STD-883A. The present investigation consisted of exposing ceramic packages sealed with these adhesives to temperature-humidity conditions of 25°C/98% RH, 50°C/60% RH, 60°C/98% RH, and 85°C/85% RH while monitoring their moisture contents using solid state moisture sensors sealed inside them. Five packages were tested at each of these exposures — two ceramic packages sealed with each of the two adhesives and one seam-sealed gold-plated Kovar package. This latter package was included to serve as a control. The solid state moisture sensor used was the Panametrics Mini-Mod-A.

The results showed that the adhesive-sealed packages were not hermetic to moisture. The rates at which moisture entered the packages increased with the severity of the exposure environments (i.e.; higher temperatures and higher moisture vapor pressures) with greater dependence on temperature than on moisture vapor pressure. The results obtained for the two adhesives indicated that Ablebond 789-1 performed approximately an order of magnitude better than Epo-Tek H77 as a package sealant. The moisture permeation even of Ablebond 789-1 was such that the moisture contents of ceramic packages sealed with it reached (or calculations indicated they would reach) 6000 ppm_v (the maximum moisture content allowed in a hybrid package by MIL-STD-883B) after the following exposures:

1 Day at 85°C/85% RH
9 Days at 60°C/98% RH
34 to 48 Days at 50°C/60% RH
236 to 332 Days at 25°C/98% RH

Unexpectedly, the results showed that the seam-sealed (brazed using gold-tin preforms) gold-plated Kovar packages also were not hermetic to moisture even though their helium leak rates were in the low-to-mid 10^{-9} atm cc/sec air equivalent region both before and after exposure to the different temperature-humidity environments. The exposure durations (either directly observed or calculated) required for the moisture contents of these packages to reach 6000 ppm_v were as follows:

1.25 Days at 85°C/85% RH
18 Days at 60°C/98% RH
17 Days at 50°C/60% RH
304 Days at 25°C/98% RH

The most reasonable explanation for this result is that expansion at the elevated temperatures causes leaks to develop that do not exist at room temperature at which the fine leak test is performed and that are not detectable during the brief 125°C exposure of the Cl gross leak test. This suggests that the present procedure of conducting the fine-leak test at room temperature and assuming that the results obtained characterizes the seal condition for the complete temperature range to which the packages subsequently are subjected is questionable. It is therefore recommended that a study be performed to investigate this situation further.

Experience with the Panametrics Mini-Mod-A moisture sensors indicated that while they are useful for the measurement of the moisture contents of packages, their calibrations shift substantially after combination exposures to cure schedules as low as 150°C for 1.5 hours and temperature-moisture content environments as experienced in this study. For such applications, the sensors must be recalibrated after the exposures and the results interpreted using these calibration curves.

4.0 REFERENCES

1. M. G. Kovac, D. Chleck and P. Goodman, "A New Moisture Sensor for In-Situ Monitoring of Sealed Packages," Solid State Technology (February 1978) Vol. 21, No. 2.
2. K. L. Perkins and J. J. Licari, "Investigation of Low Cost, High Reliability Sealing Techniques for Hybrid Microcircuits," Phase I, Final Report, April 1976; NASA Contractor Report NASA CR-144339.